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Dr. Shawn H. Phillips

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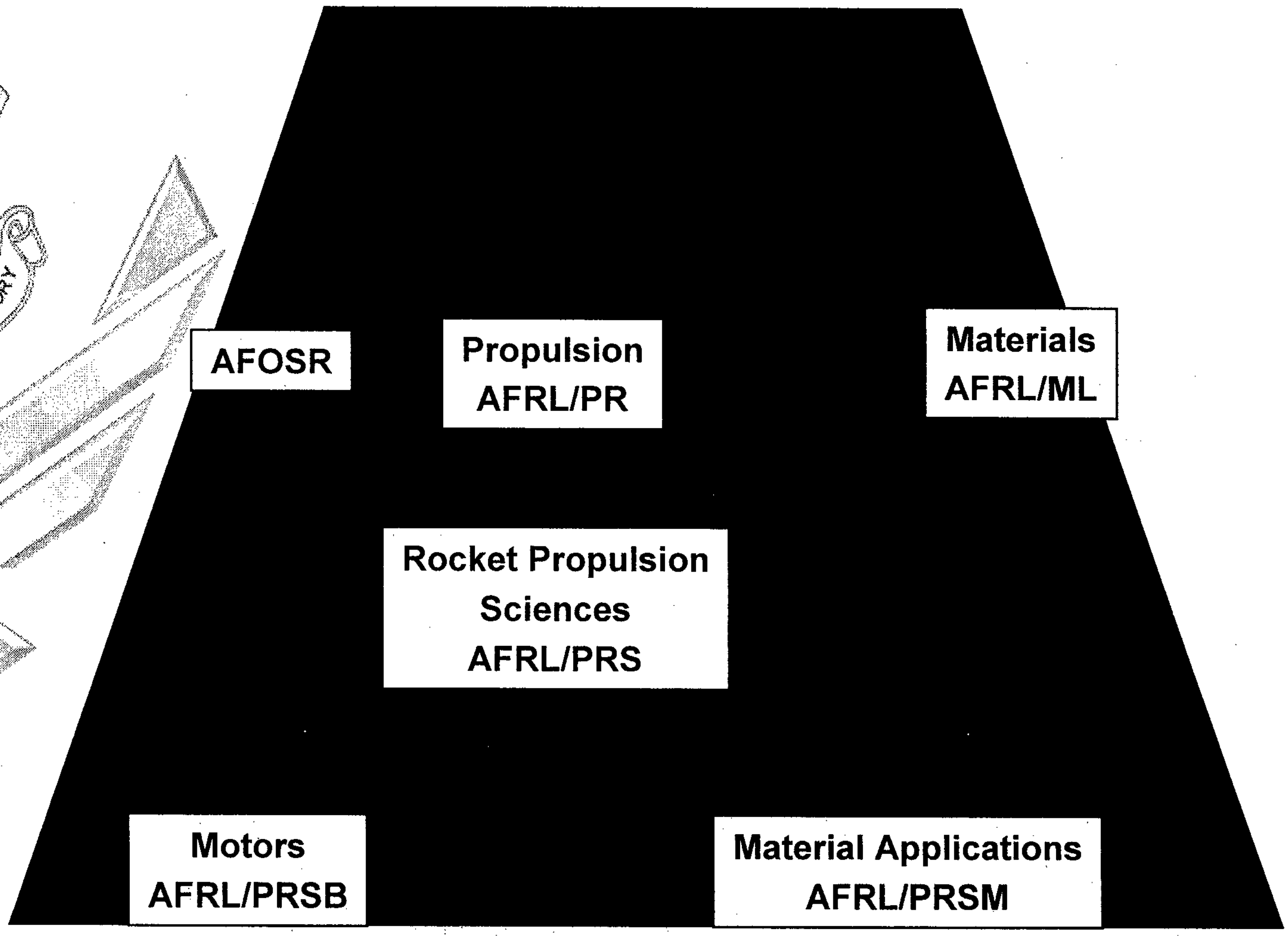
Materials Applications Research within the Propulsion Directorate of the Air Force Research Laboratory (AFRL/PRSM)



"Seated within the Propulsion Directorate of the Air Force Research Laboratory, the Material Applications Branch has as its Mission to Apply and Transition Materials Technology to Rocket Propulsion."

Dr. Shawn H. Phillips
Chief, AFRL/PRSM
Air Force Research Lab, Edwards

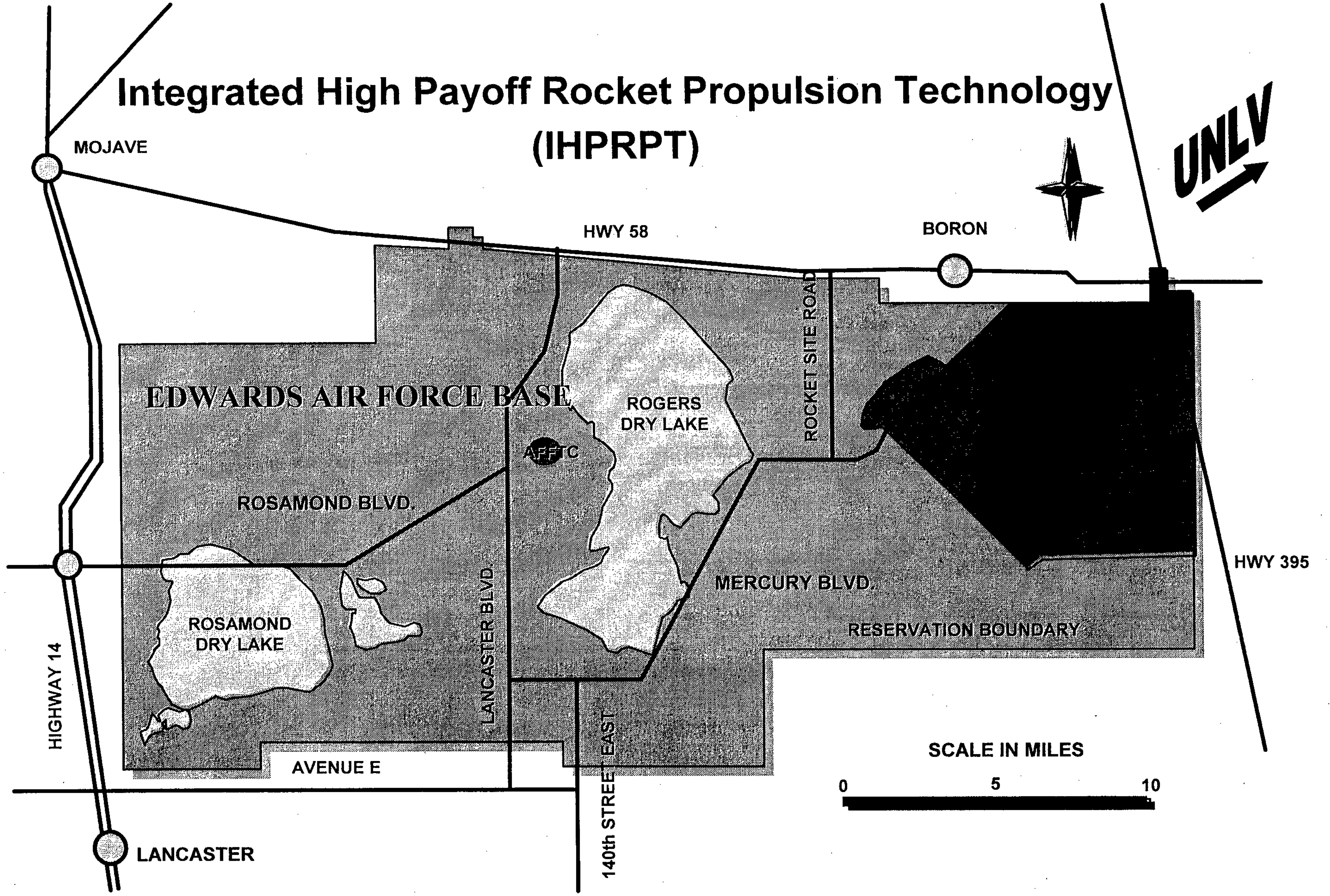
High Temperature Components Group

Polymer Working Group

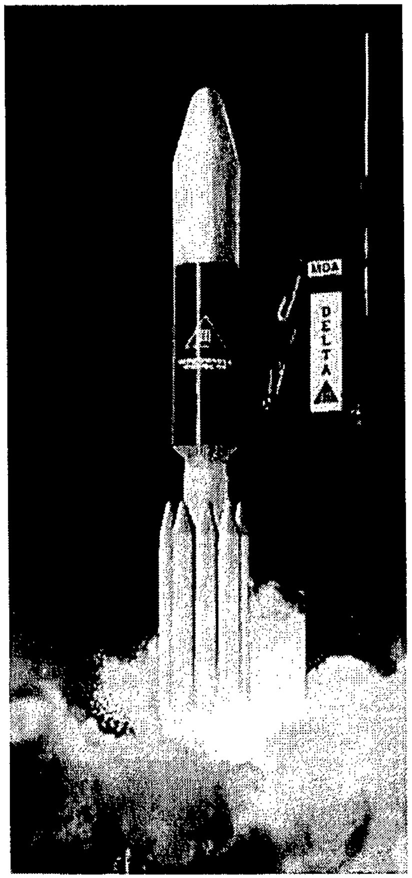
Fracture Mechanics Group

Air Force Research Laboratory (Edwards)

Integrated High Payoff Rocket Propulsion Technology (IHPRPT)



Research Focus within AFRL/PRSM



High Temperature Components

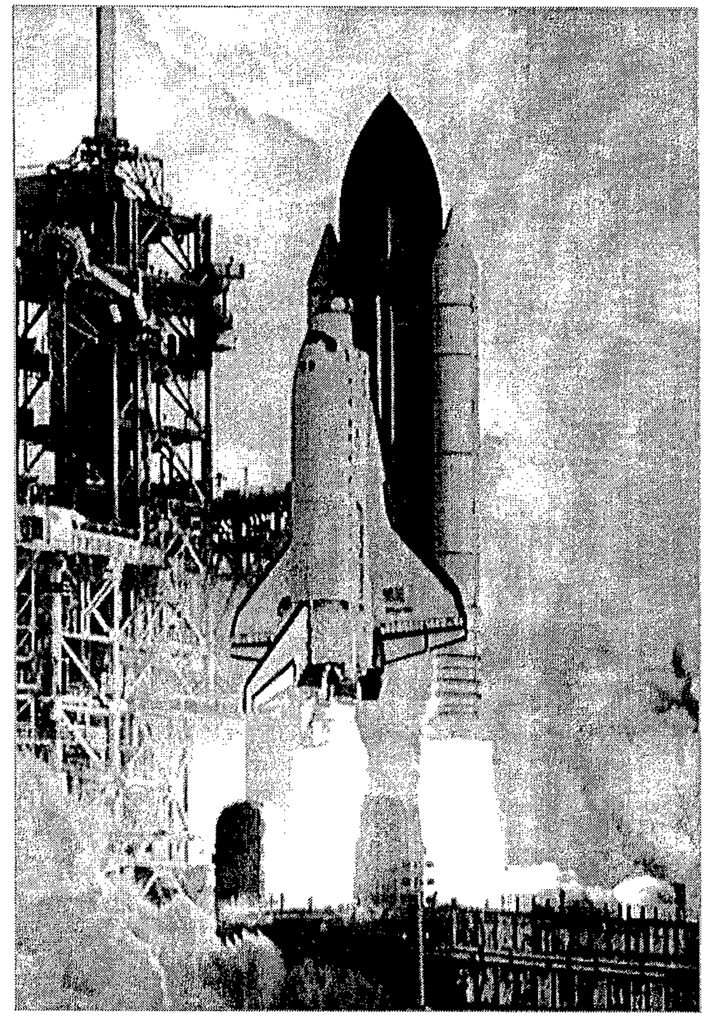
- Rapid Densification of Carbon-Carbon
- Microtube Technology
- Scale-up and Commercialization of Rapid Densification of Carbon-Carbon

POSS Nanostructured Polymers

- Structure/Property Relationships
- Thermo/Mechanical Improvement of Polymers
- Space-survivable Materials and Coatings
- Scale-up and Commercialization of POSS Nanotechnology

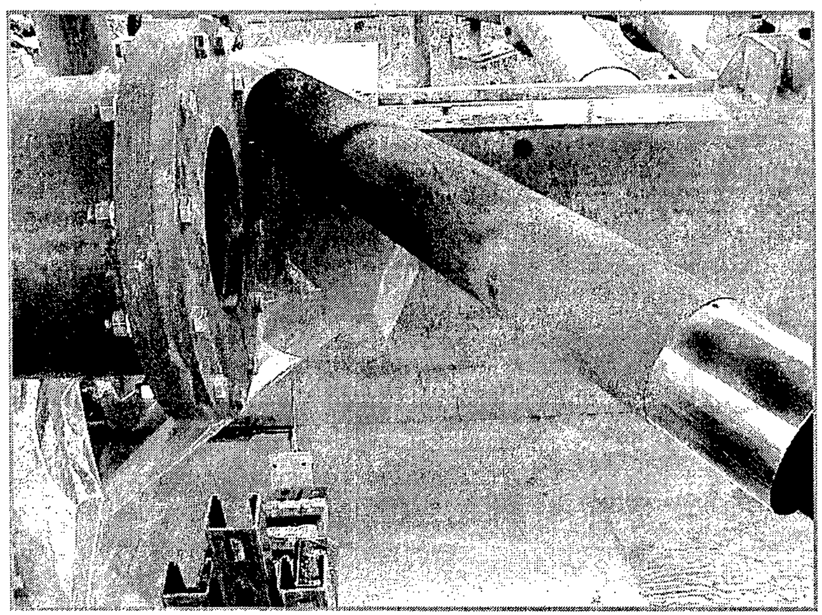
Rapid Densification of Carbon-Carbon

- Carbon-Carbon Advantages
  - Excellent High Temperature Structural Material
  - Very Reliable in Rocket Nozzles, Exit Cones, Nosetips, and Leading Edges As Well As Aircraft Brakes
- Drawbacks to Carbon-Carbon
  - SOTA Production of Carbon-Carbon Is Very Expensive
  - Carbon-Carbon Oxidizes at High Temperature in the Presence of Oxidizers



Objectives

- Decrease the processing time of Carbon-Carbon composites from many months to less than two weeks.
- Cut the densification cost in half.



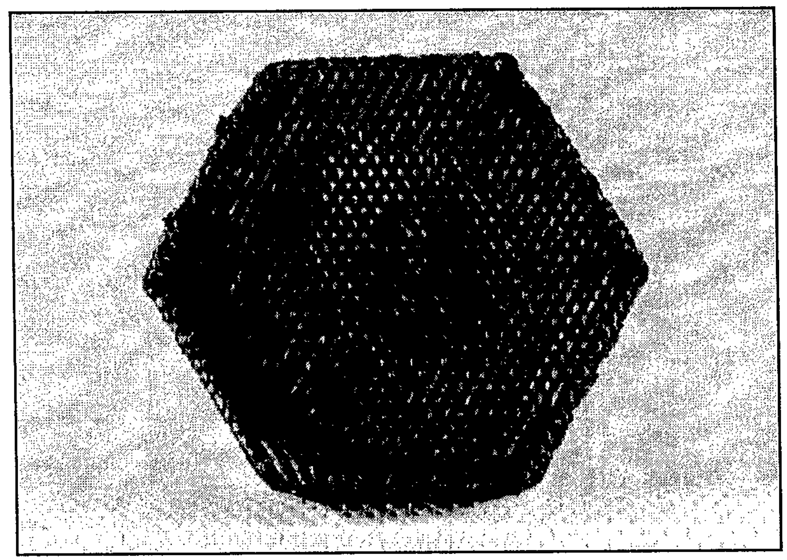
Carbon-Carbon part densified in less than two weeks

Rapid Densification of Carbon-Carbon

Technical Challenge:

- With Conventional Liquid Phase Processes There Is Incomplete Penetration of the Liquids Due To:

  a.) High Viscosity
  b.) High Surface Tension
  c.) Gassing of Precursor



- With Gas Phase Processes There Is Incomplete Penetration of the Gases Due to Their Decomposition on the Outer Surface

Rapid Densification of Carbon-Carbon

Technical Approach:

- For Liquid Processes Use an Impregnant That Has:
  - a.) A High Carbon to Hydrogen Ratio (Char Yield)
  - b.) Low Viscosity
  - c.) Wets the Fiber Preform.
- In Normal Processing It Is Impossible to Get This Combination of Properties
  - High Char Yield Needs High Molecular Weight
  - Low Viscosity and Wetting Require Low Molecular Weight

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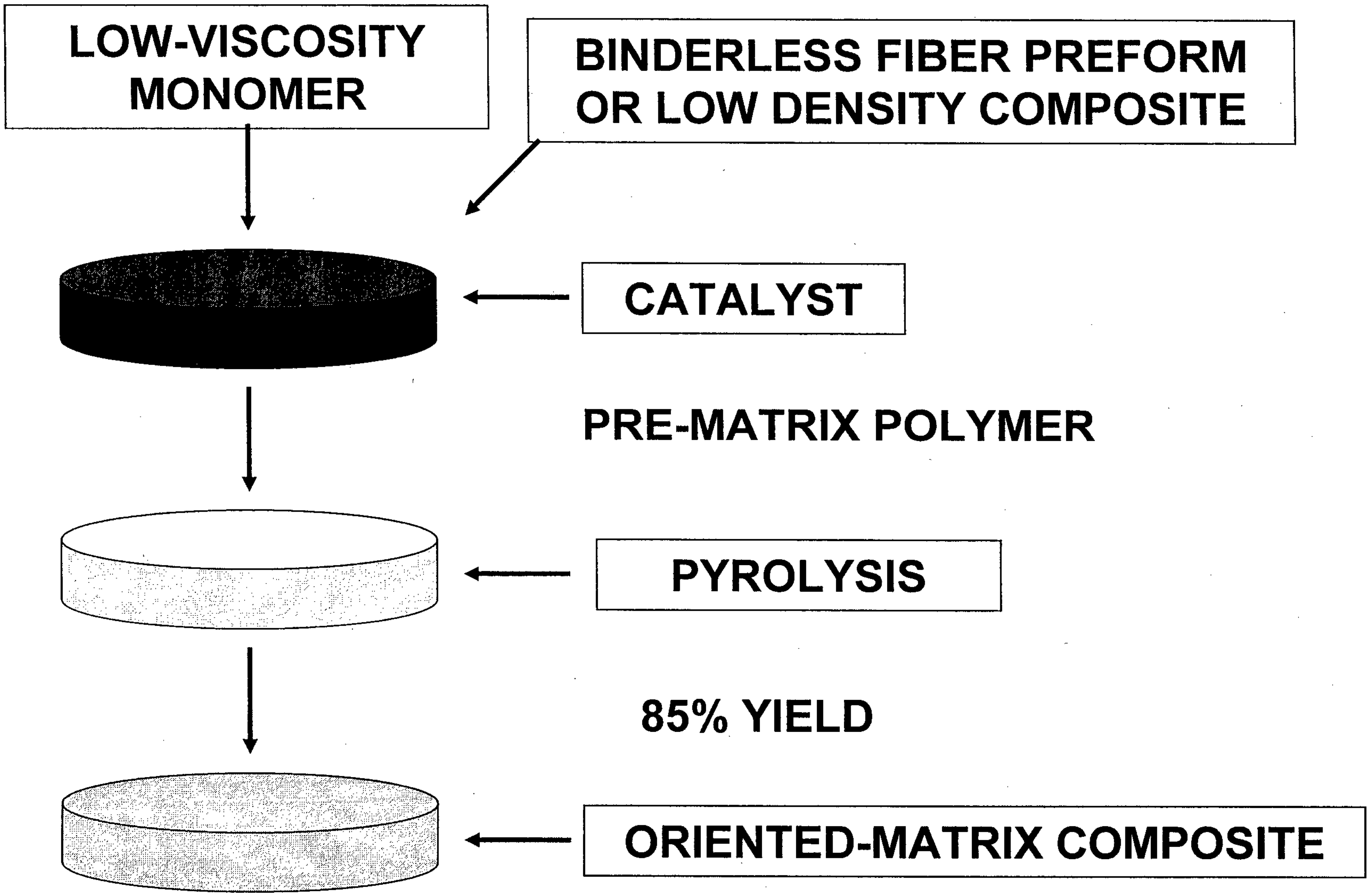
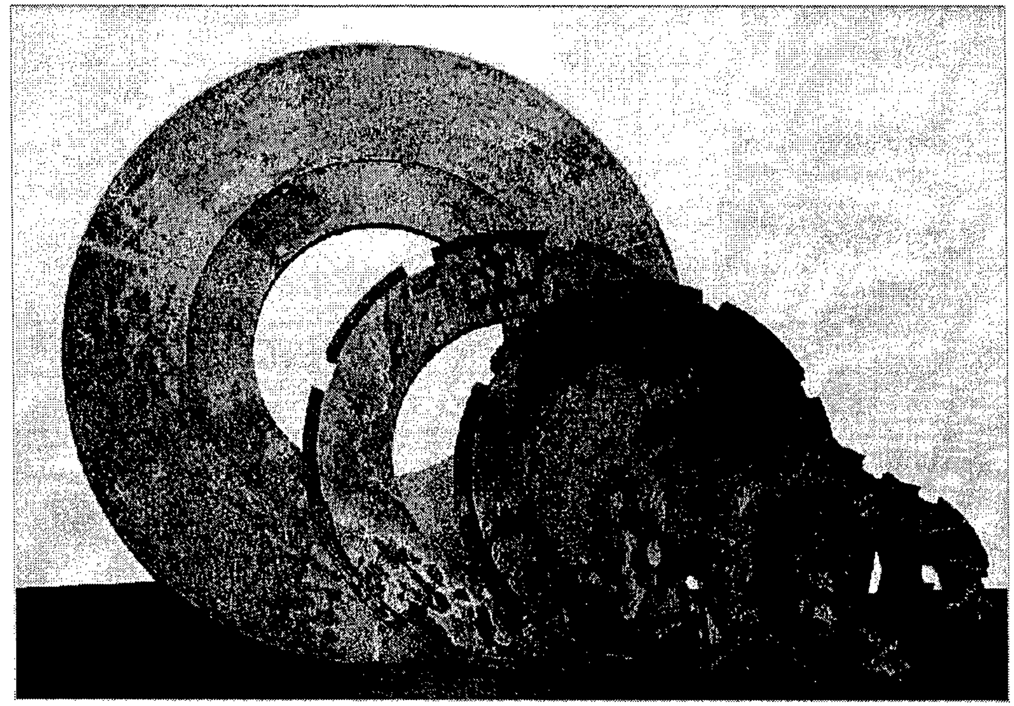
In-Situ Formation of Carbon and Ceramic Matrices



Process Advantages

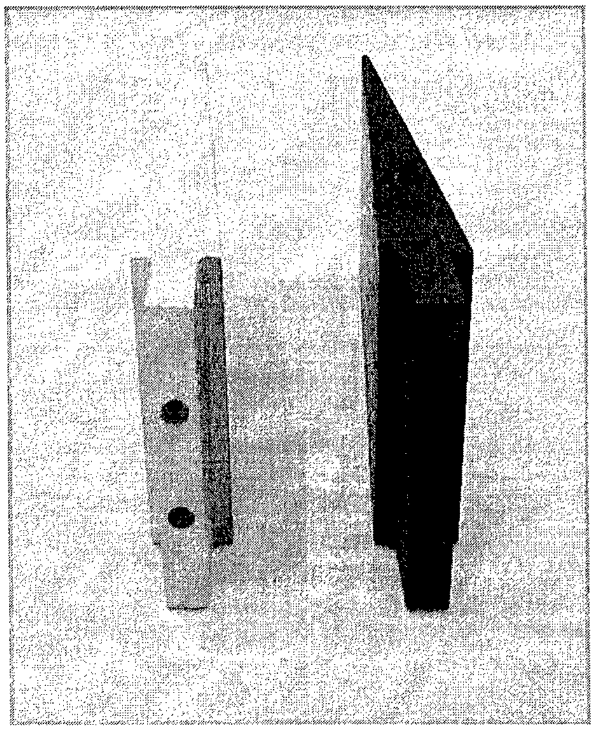
- Very Uniform Density
- Can Densify Thick Composite
- Complex Geometries
- No Need to Graphitize
- No Need to Machine Between Densification Cycles



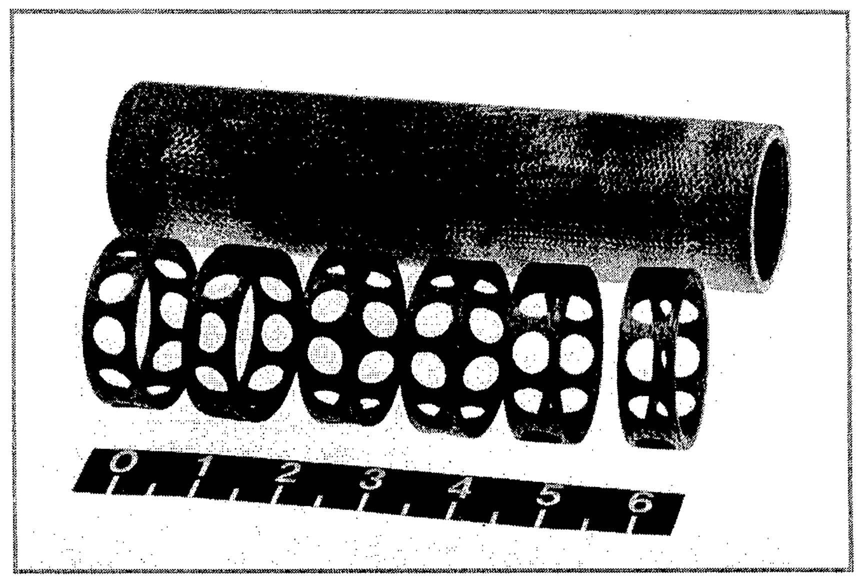
Compatible With Vapor-grown High-conductivity Fibers

- Dual Use: Carbon-Carbon Brakes and Electronic Thermal Management

Accomplishments



- Large Reactors have been Designed….Installed Allowing Scale-up to 18" Diameter and 60" Length.
- A Large (10" X 10" X 8") Preform and a 5' X 8" Tube Were Uniformly Densified With High Quality Matrix in 2 Weeks. (Not Possible With Other Processes)



- Technology Transfer to SMJ Carbon is ongoing with a cooperative research agreement.

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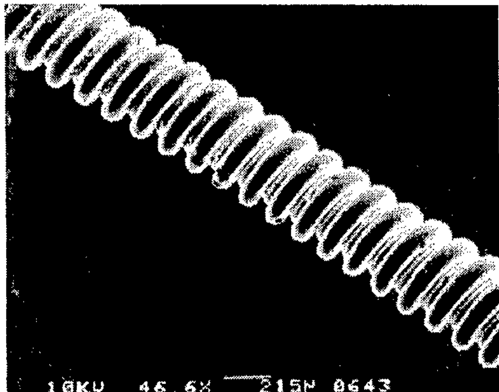
Microdevice Fabrication and Micropropulsion



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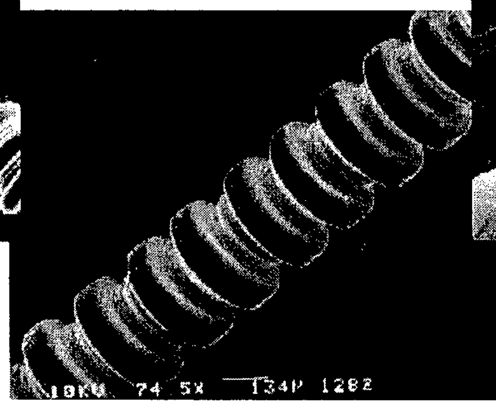

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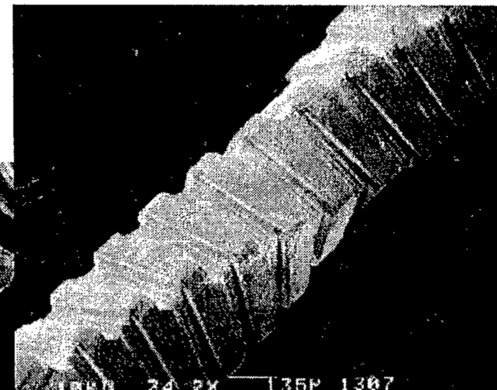

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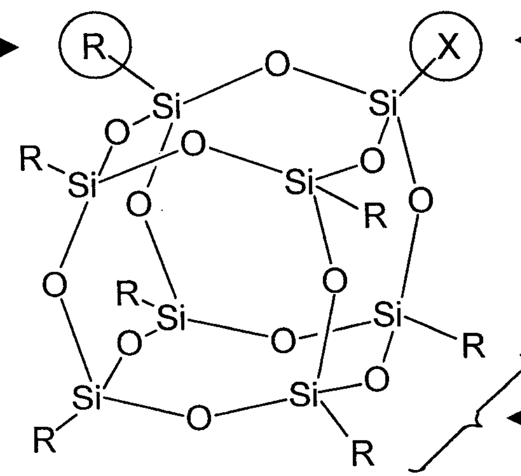
- Revolutionary method can make any 3-D micron scale shape from any material--1st reliable 3-D manufacturing method
- Heat exchangers, sensors, ducts and valves have all been successfully made using this process

Anatomy of a Polyhedral Oligomeric Silsesquioxane (POSS®) Molecule

Nonreactive organic (R) groups for solubilization and compatibilization.

May possess one or more functional groups suitable for polymerization or grafting.



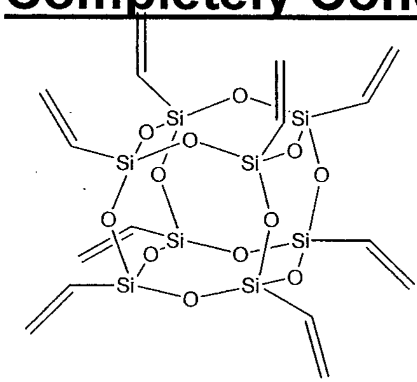
Nanoscopic in size with an Si-Si distance of 0.5 nm and a R-R distance of 1.5 nm.

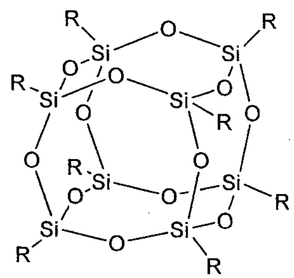
Thermally and chemically robust hybrid (organic-inorganic) framework.

Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.

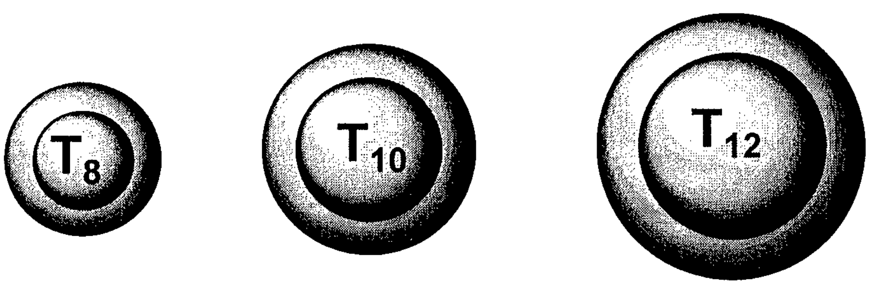
POSS®

Completely Condensed

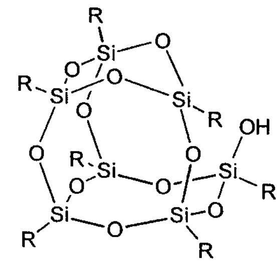




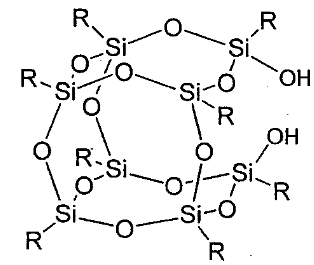
R = Me, Et, *i*-Bu, Cp, Cy, i-Octyl, Ph



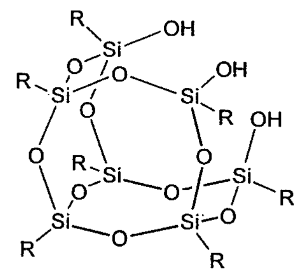
Incompletely Condensed



R = Cp, Cy, *i*-Bu



R = Cy, Cp, i-Bu, Et

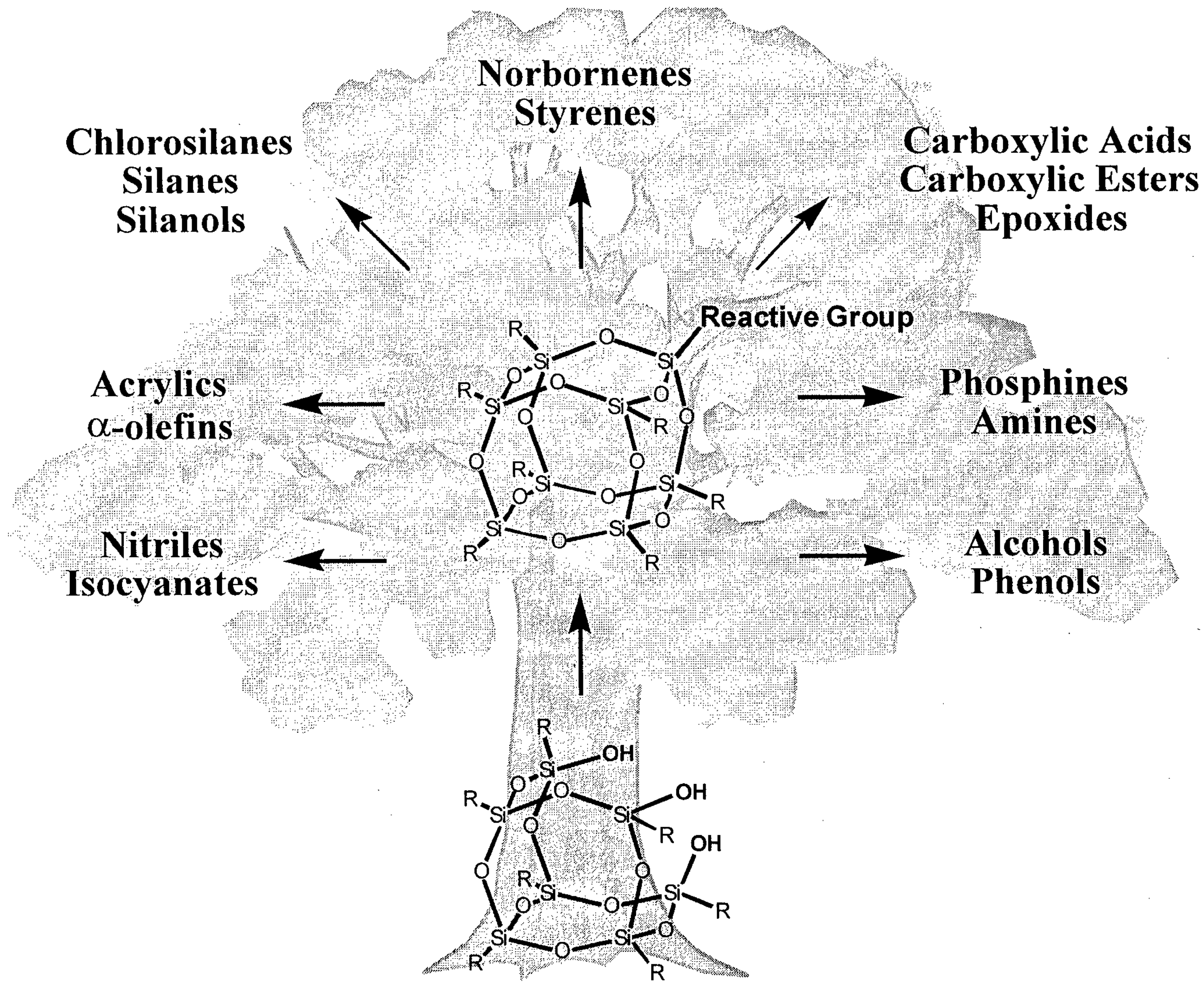


R = *i*-Butyl, Et

>180 POSS Monomers are commercially available!!

www.hybridplastics.com

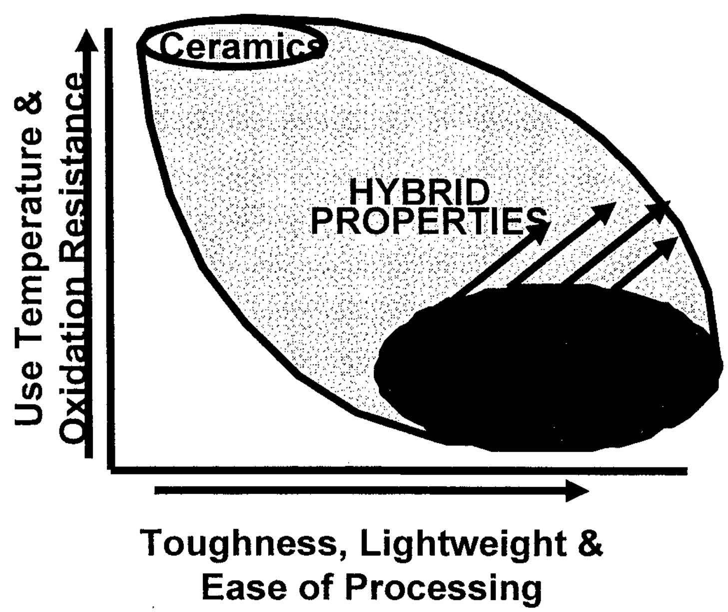
Functionalized POSS®-Monomers



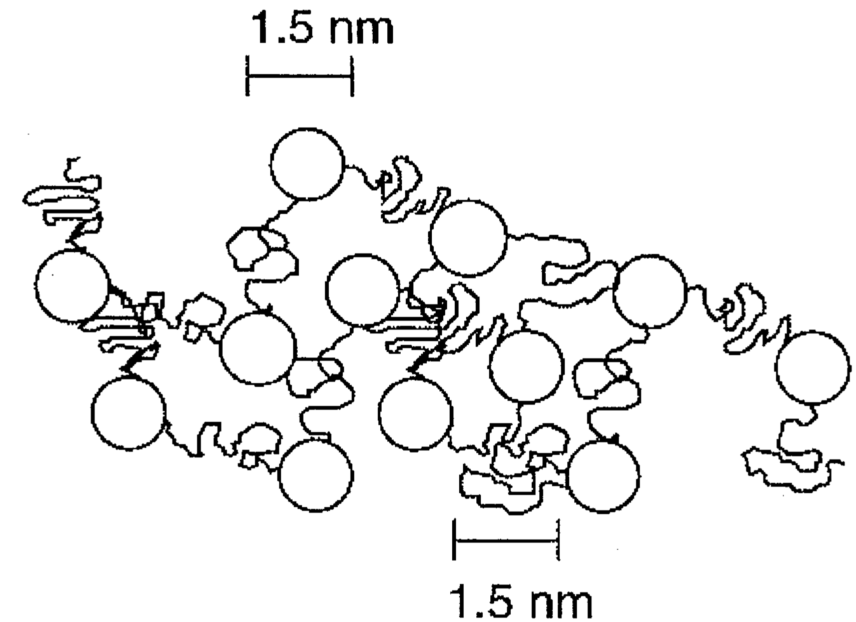
Hybrid Plastics currently offers over 180 Nanostructured™ Chemicals

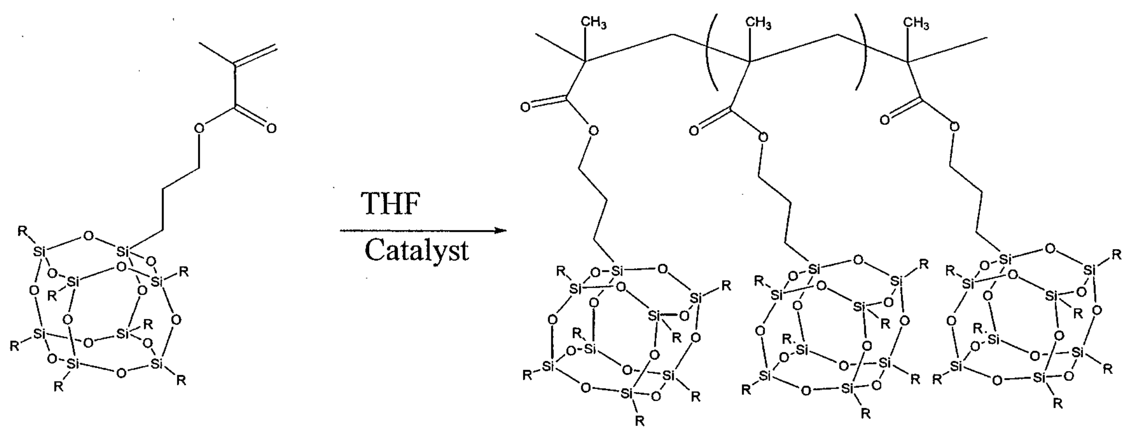
Key Aspects of POSS® Technology

Hybrid (inorganic/organic) Composition



Nanostructured™ Chemical Reinforcement

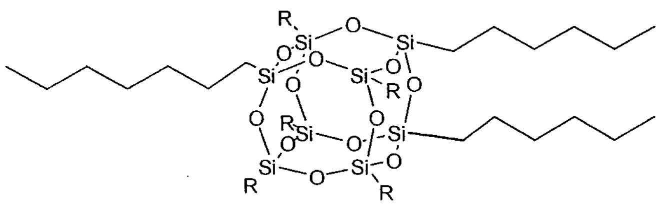




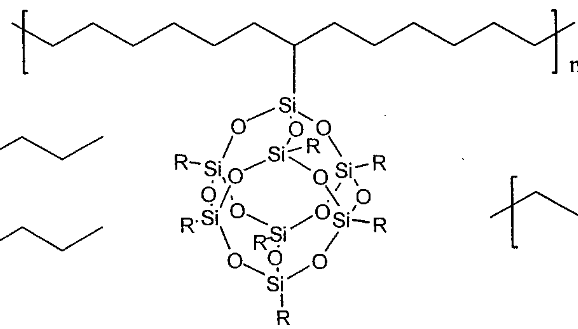
POSS™ technology does not require manufacturers to retool or alter existing processes.

Lichtenhan et. al. *Macromolecules* **1993**, *26*, 2141.
Lichtenhan, *Polym. Mater. Encyclopedia* **1996**, *10*, 7768.

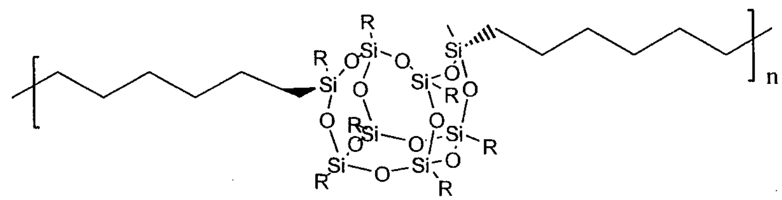
POSS® Polymer Incorporation



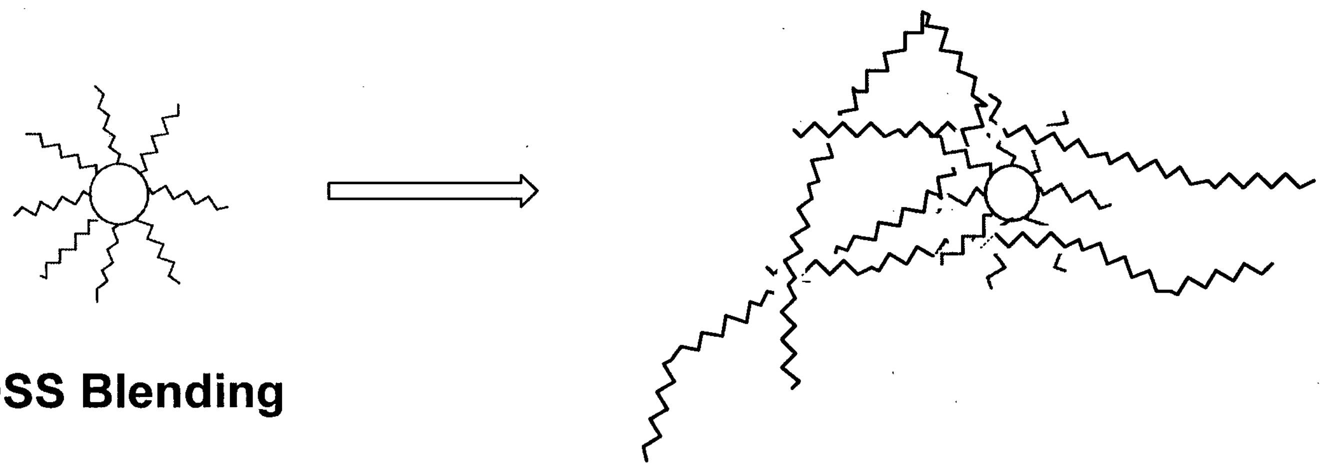
Cross-linker



Pendant Polymer

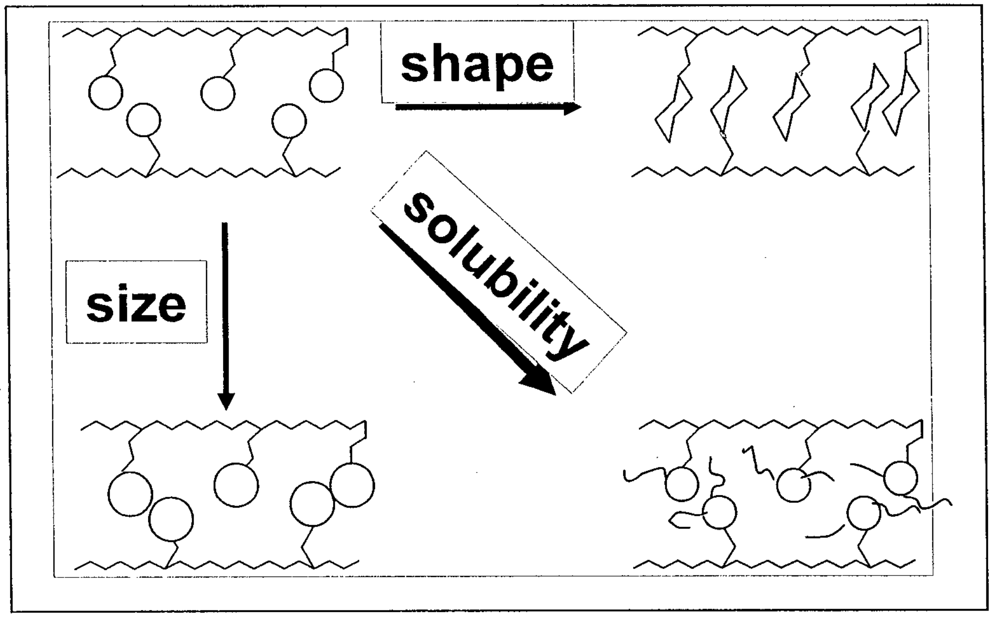


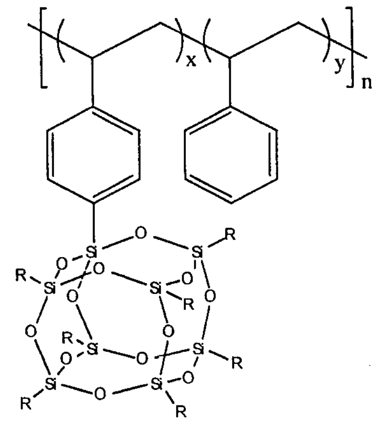
Bead Copolymer

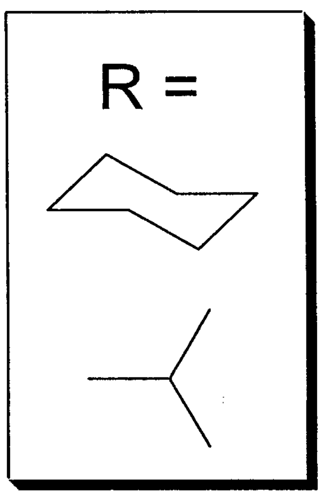


POSS Blending

Structure/Property Relationships

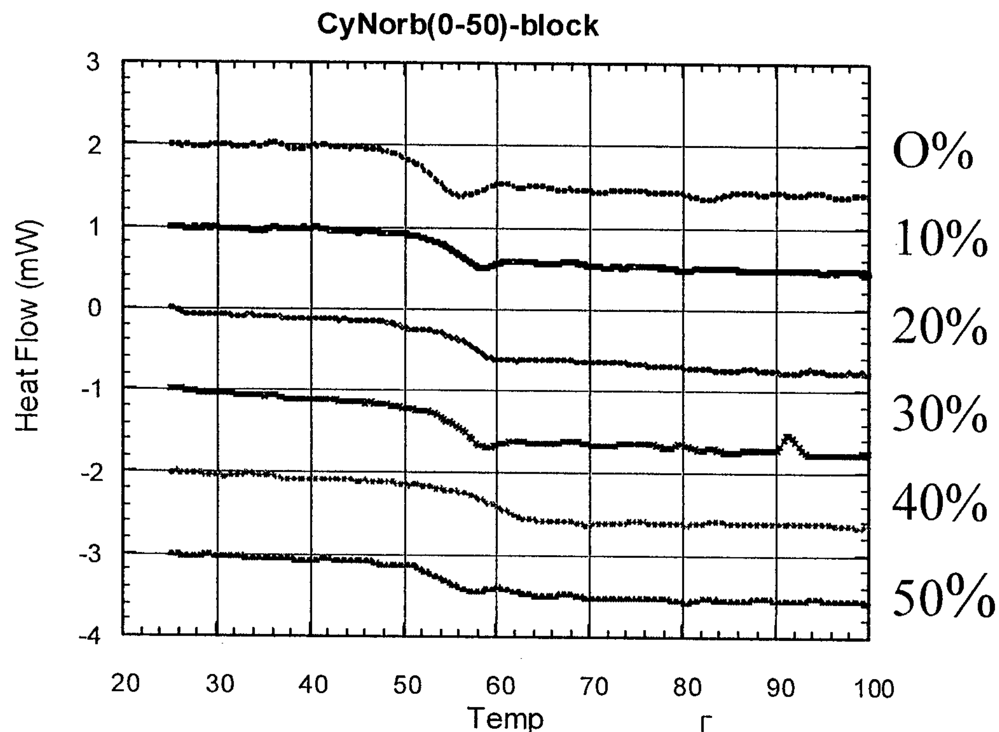


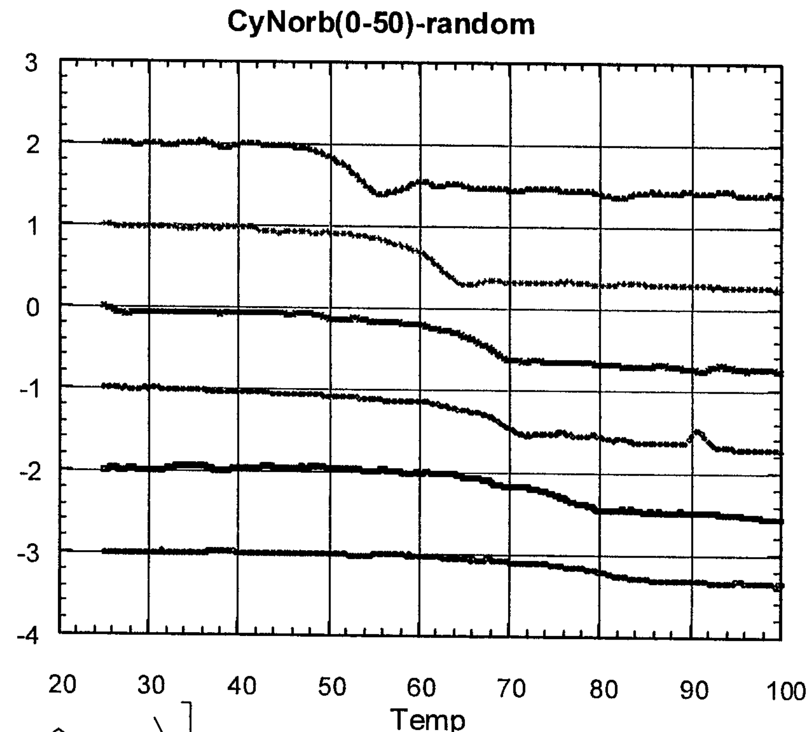


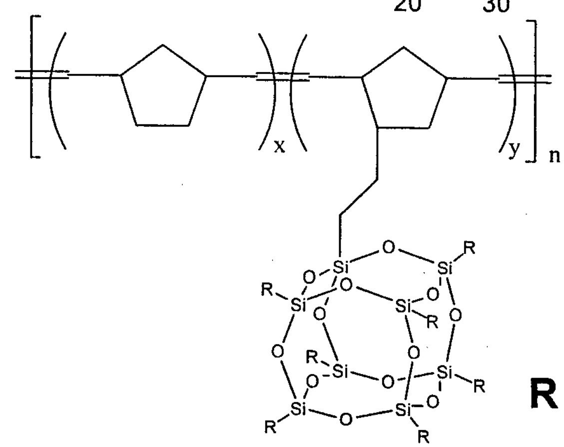


- Maximizing property enhancements through changes at the nano level
- Polymer compatibility vs. POSS/POSS interactions

DSC Data for POSS®-Norbornenes

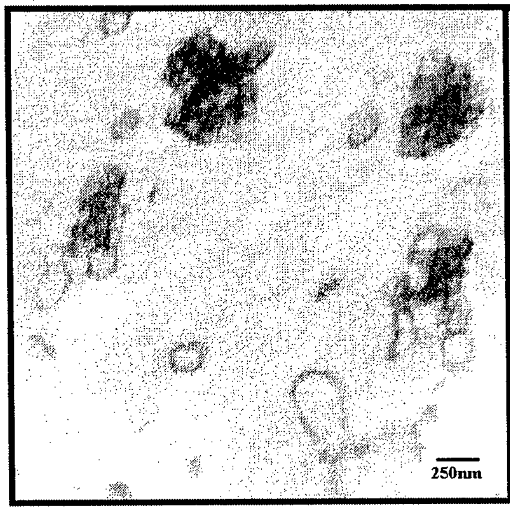






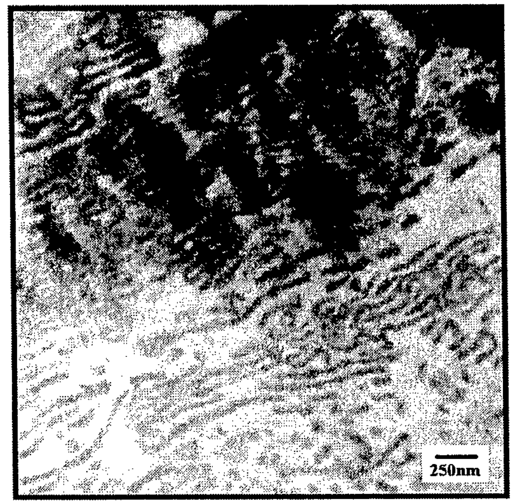
R = Cyclohexyl or Cylcopentyl

TEM of Diblock POSS®-Norbornenes

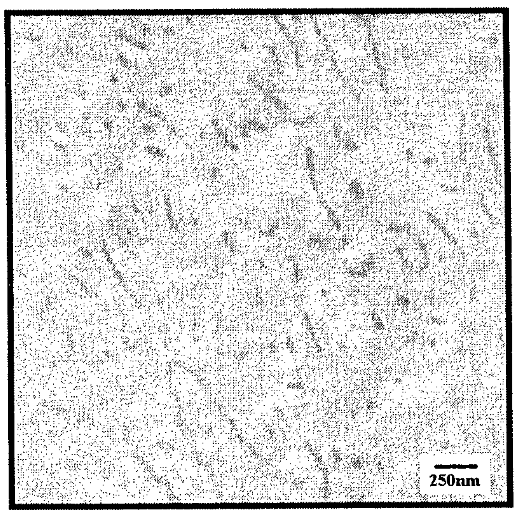


10wt % of CpPOSS

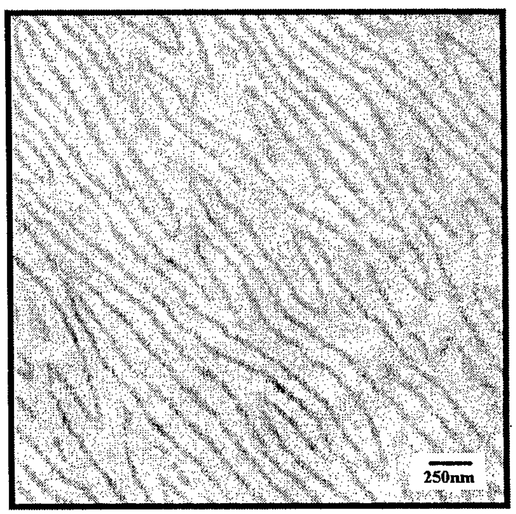
30wt% of CpPOSS



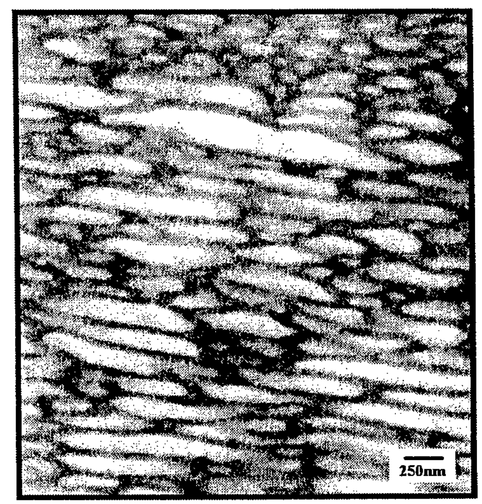
60wt% of CpPOSS



10wt% of CyPOSS



30wt % of CyPOSS

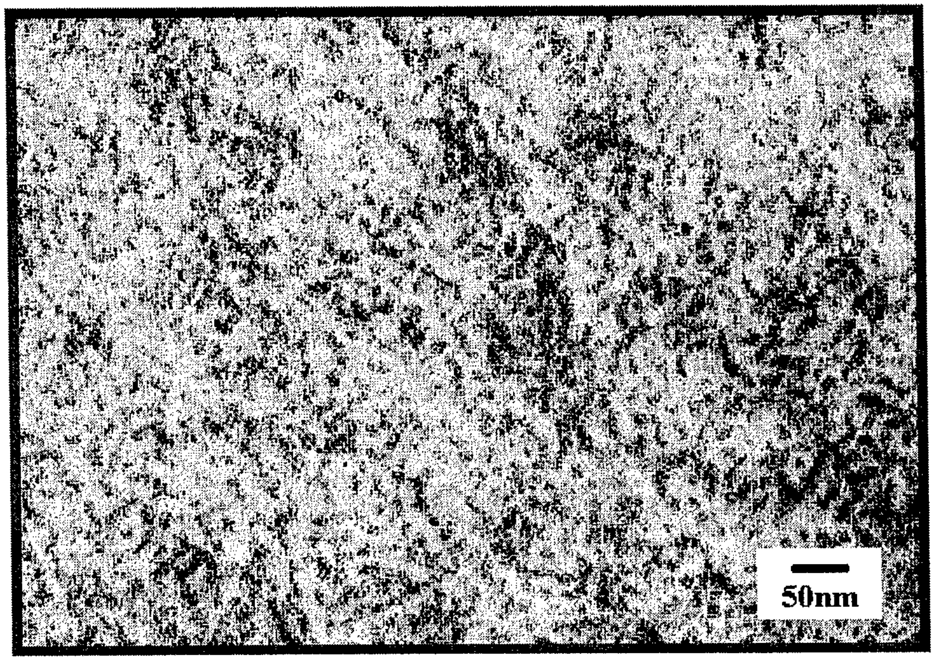


60wt% of CyPOSS

Pat Mather, AFRL

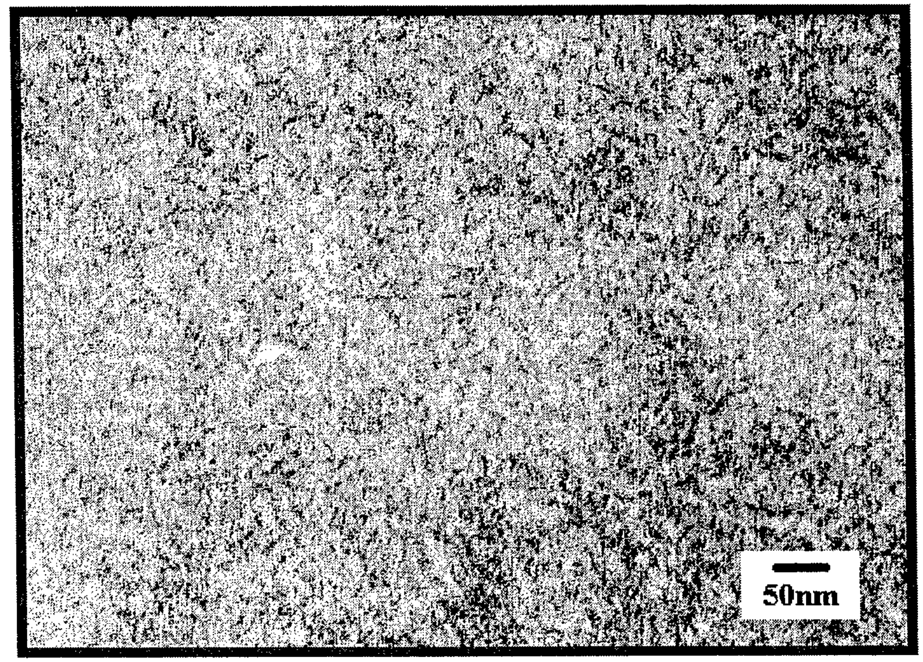
TEM of Random POSS®-Norbornenes

50CyPOSS/PN



"Coarse" Cylinder Nanostructure
(Diameter ~ 12nm)

50CpPOSS/PN



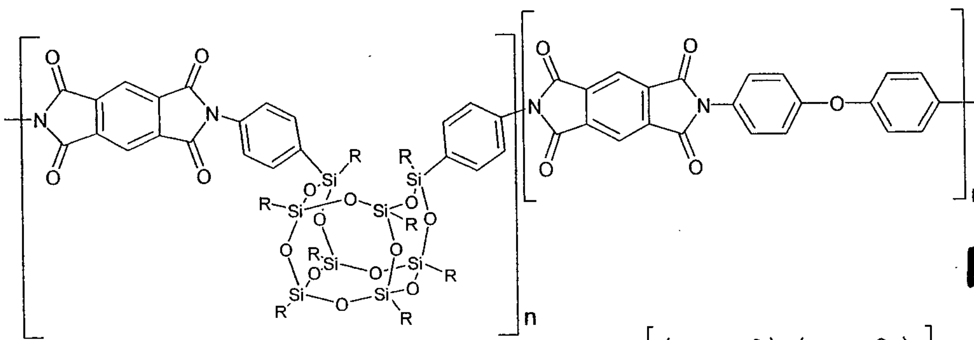
"Fine" Cylinder Nanodstructure
(Diameter ~ 6nm)

Since the glass transition improvement is almost double when R=cyclohexyl, then cyclohexyl POSS-rich domains may entrain more unoriented polynorbornene chains than Cyclopentyl POSS-rich domains.

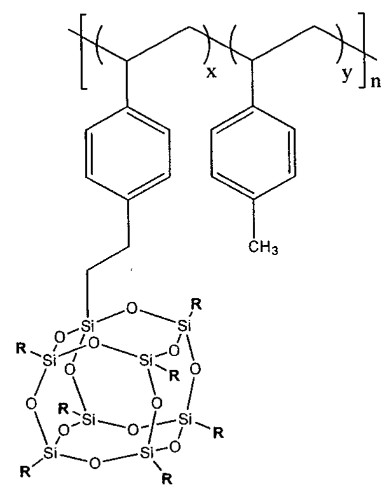
Pat Mather, AFRL

Thermo/Mechanical Improvement of Polymers

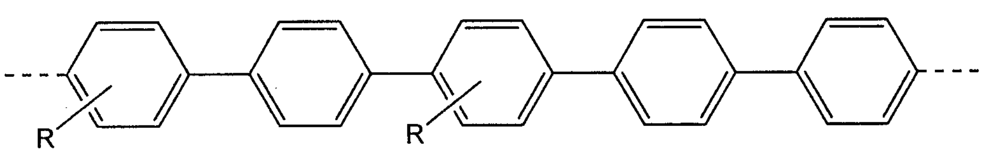
[redacted]



POSS®-Kapton® (polyimide)



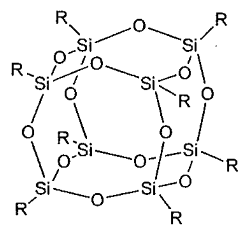
POSS®-Styrenes



Maxdem's PARMAX™ polymer

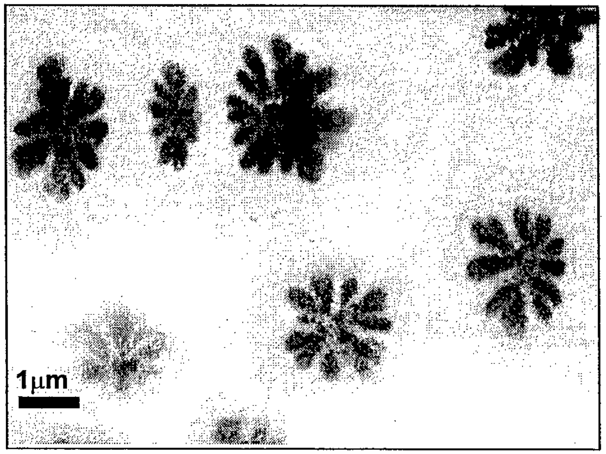
Importance of R groups: Affect compatibility with polymer matrix

50 Wt % POSS Blends in 2 Million MW PS

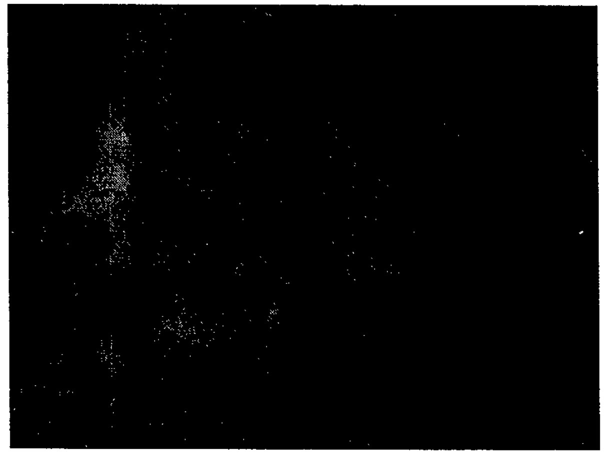


R = cyclopentyl

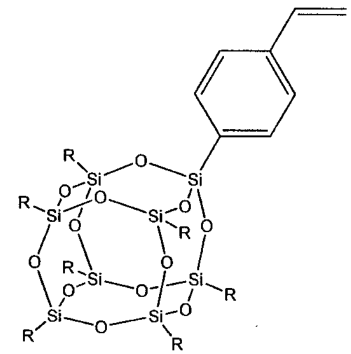
Cp_8T_8



Domain Formation

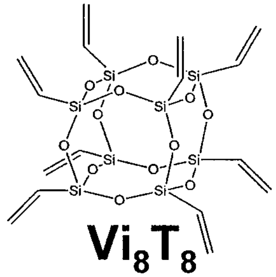


Partial Compatibility

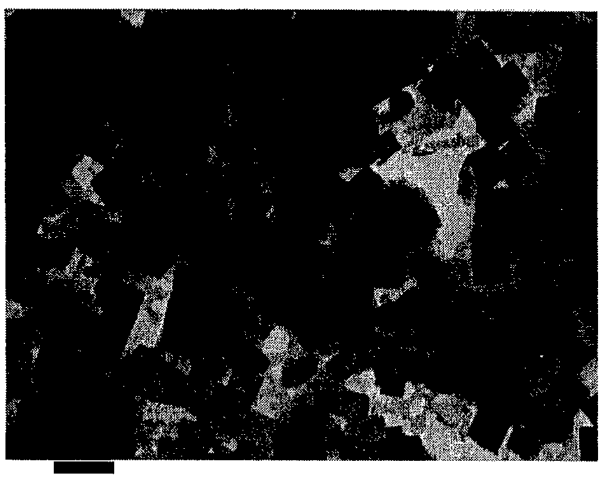


R = cyclopentyl

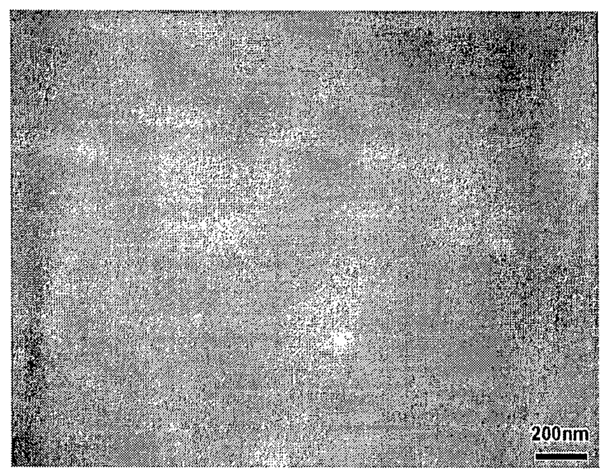
Cp_7T_8Styryl



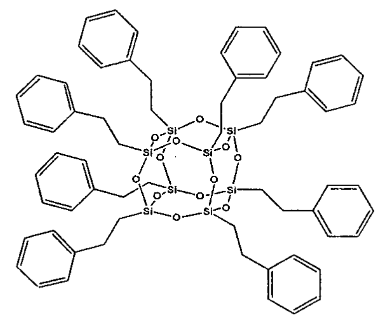
Vi_8T_8



Immiscible POSS Crystallites



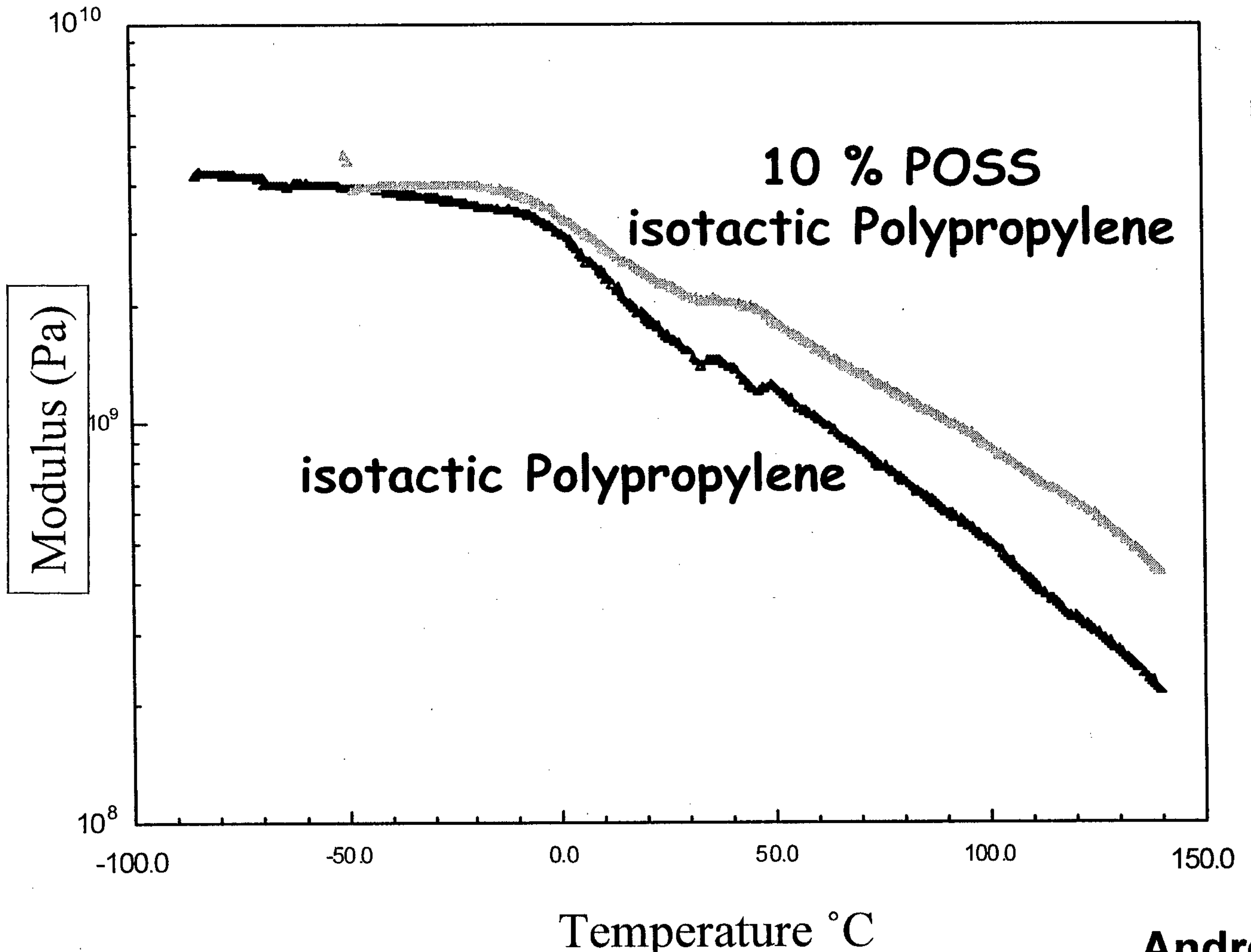
Complete Compatibility-
POSS Nanodispersion/Transparent

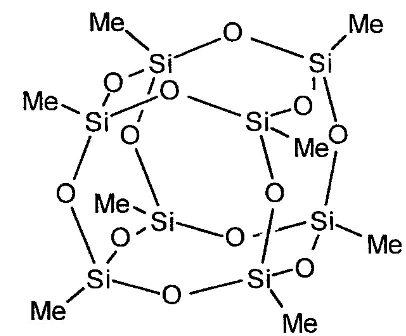


Phenethyl$_8T_8$

Blanski, et al., *Polymer Preprints*, **2000**. 41(1): p. 585.

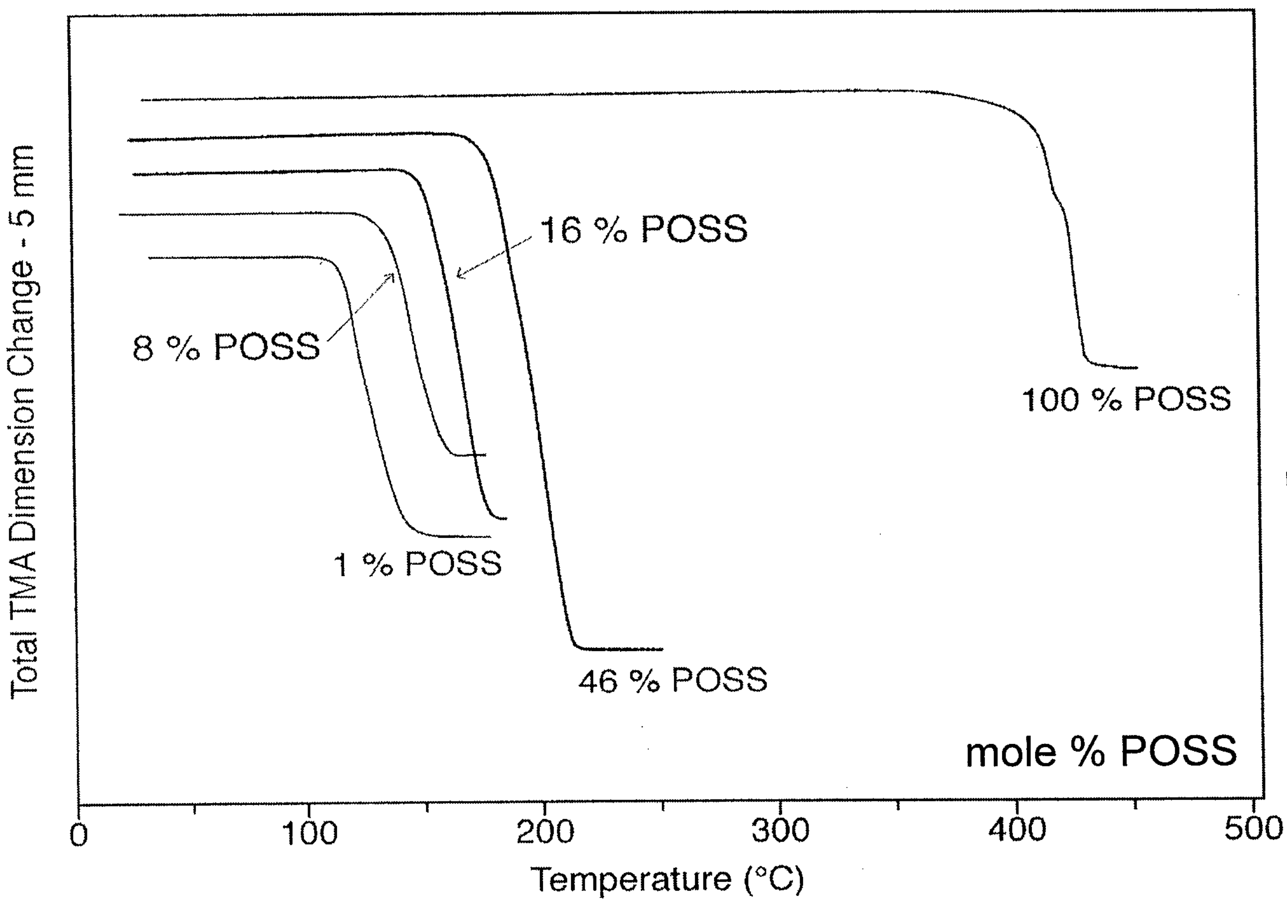
DMA of 10 Wt% POSS® in isotactic Polypropylene

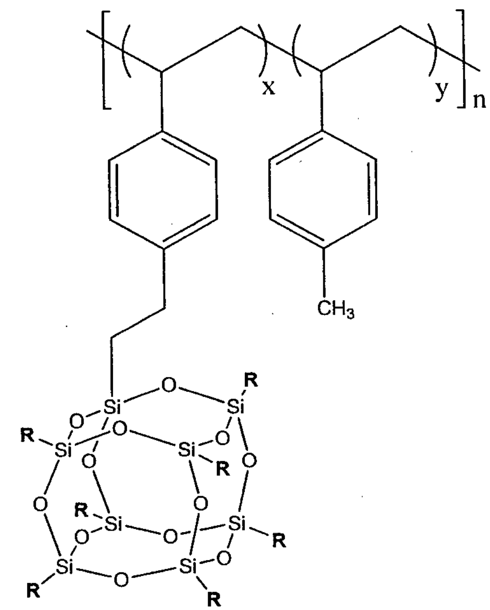




Andre Lee, AFRL

TMA Plot For POSS® Styrenes (R = Cyclohexyl)

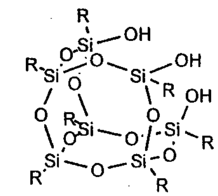


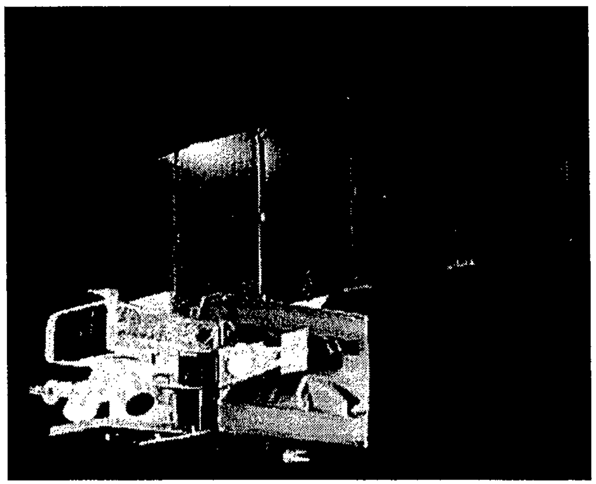


$R = C_6H_{11}$

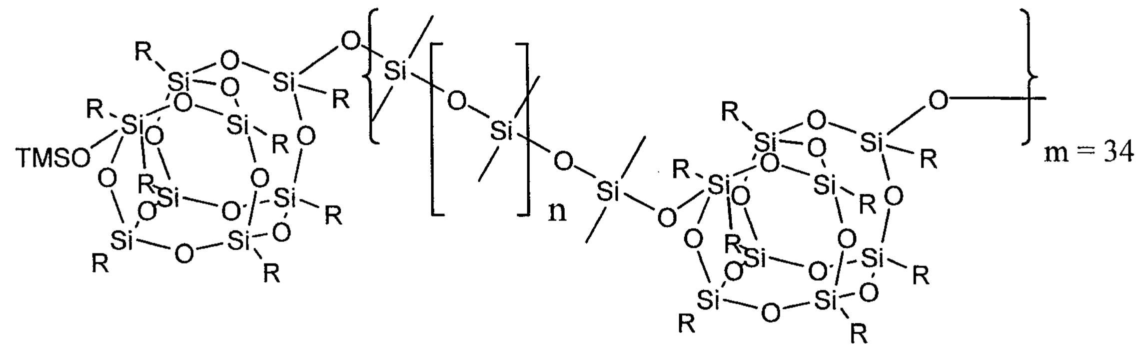
POSS® Materials for Space

Critical for Increasing Lifetime





Satellites & Space Systems



POSS®-PDMS copolymers

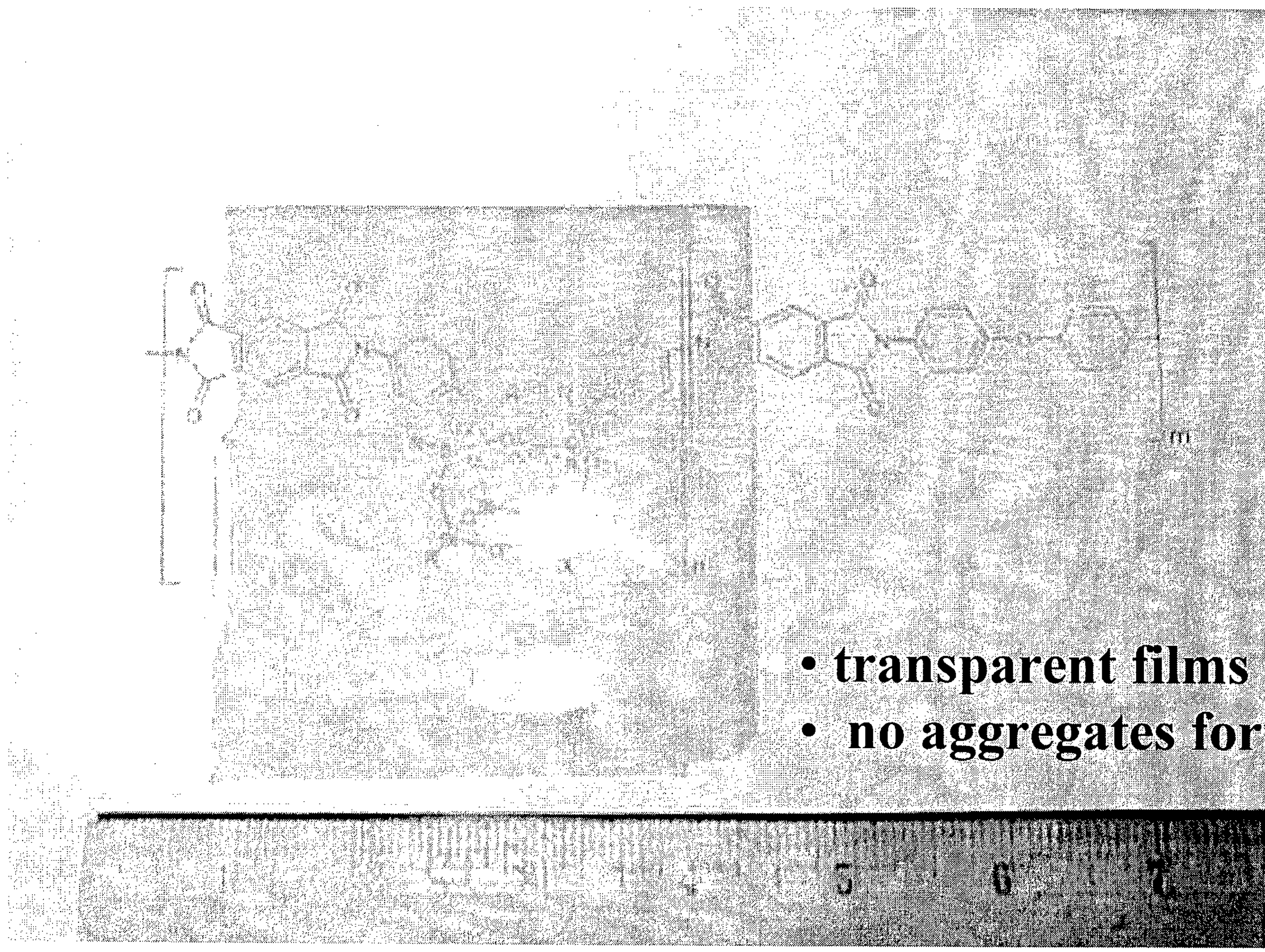
POSS Nanocomposite Payoffs

- Maximum Space Survivability
  - LEO, AO, VUV, Impact
- Lower Density 'Filler'
- High Modulus
- Resins for all Structural Applications

Simulated 3 mo. AO/VUV Exposure

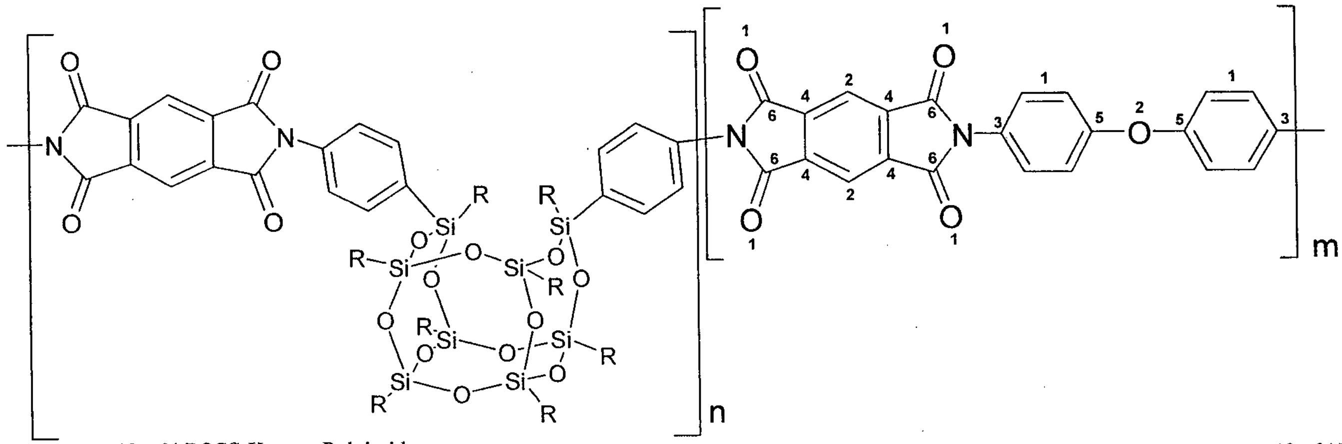
- 9-20x greater AO resistance than current state of art
- Even better AO/VUV resistance
- Passivation layer demonstrated

POSS®-Kapton® Polyimides

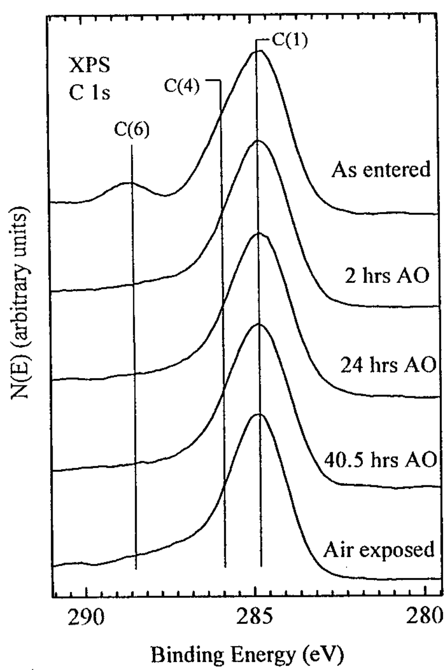


- transparent films
- no aggregates formed

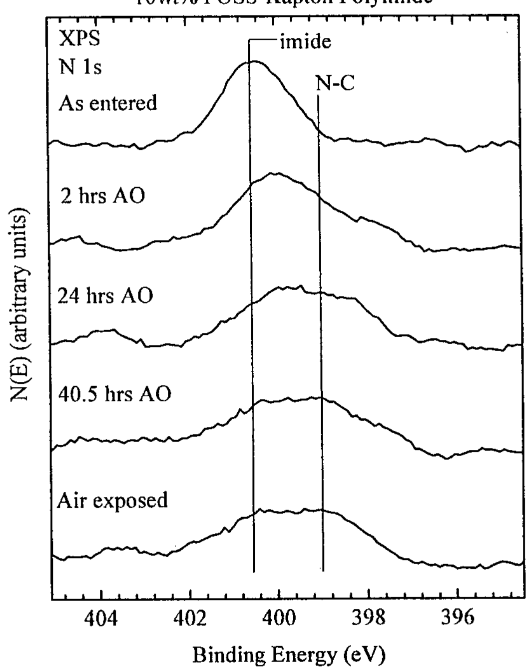
Space-Survivable POSS®-polymers



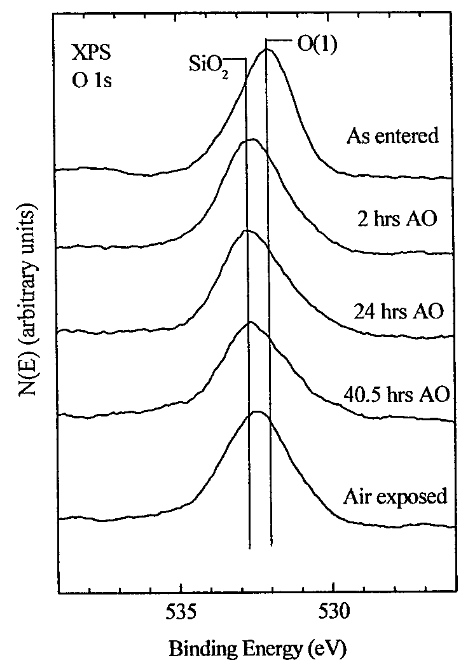
10wt% POSS-Kapton Polyimide



10wt% POSS-Kapton Polyimide

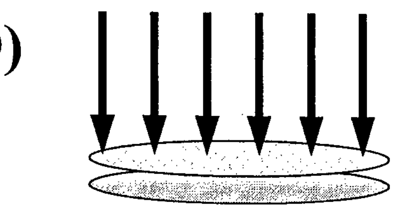


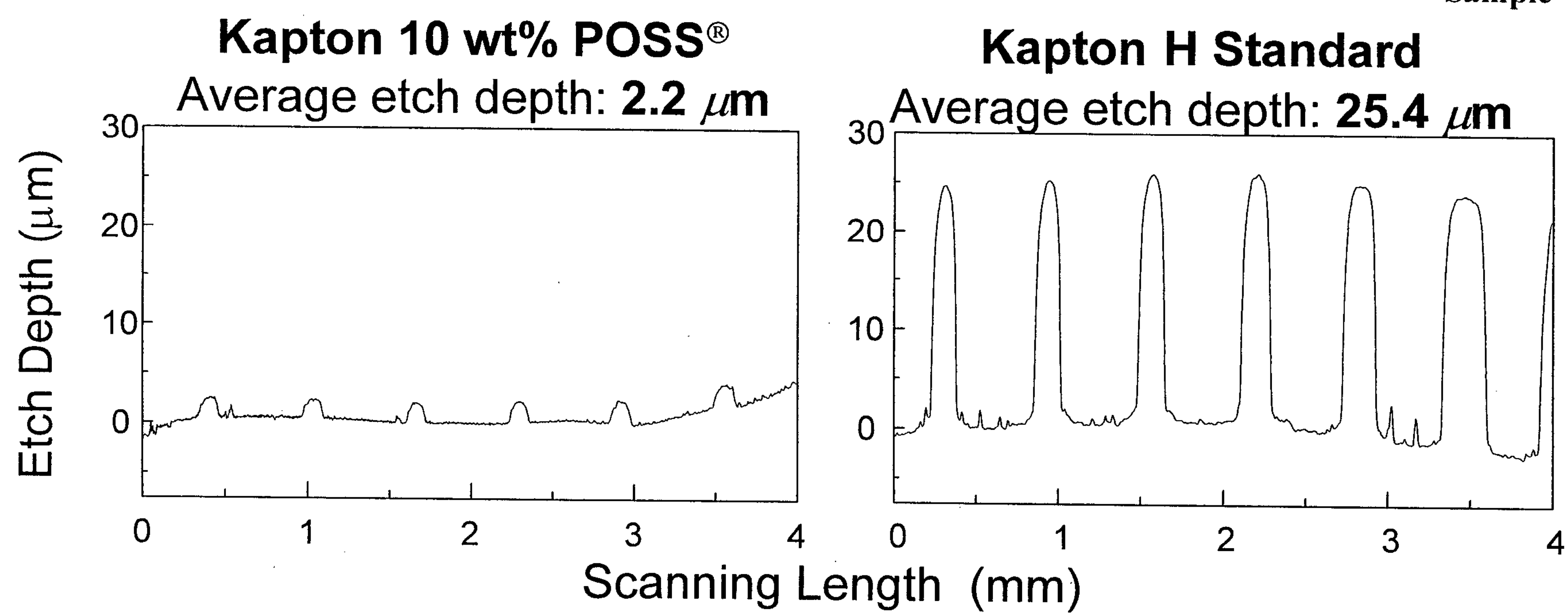
10wt% POSS-Kapton Polyimide



Independent Verification of Oxidation Resistance

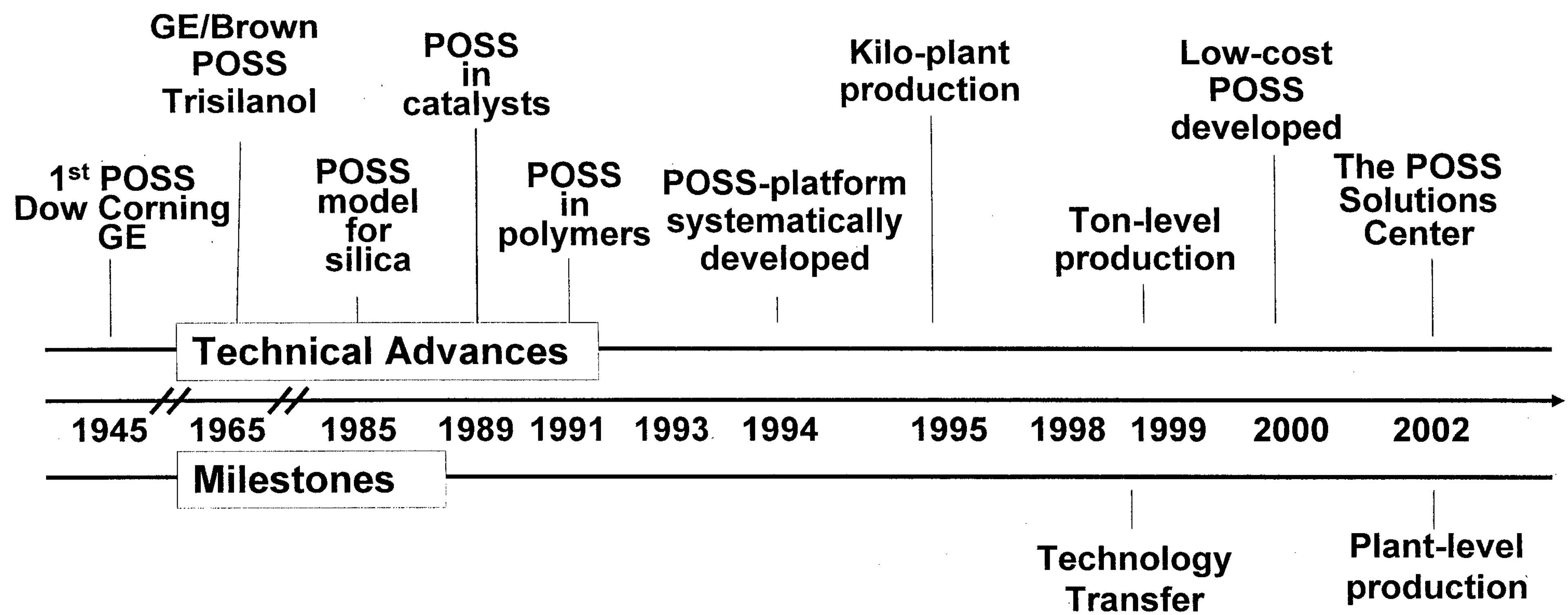
O-Atom Etching Experiment (~10 DAYS IN LEO)
Total AO fluence of 8.47 x 1020 atoms cm-2





Significantly improved oxidation resistance due to a rapidly formed, ceramic-like, passivating and **self-healing** silica layer preventing further degradation of underlying virgin polymer.

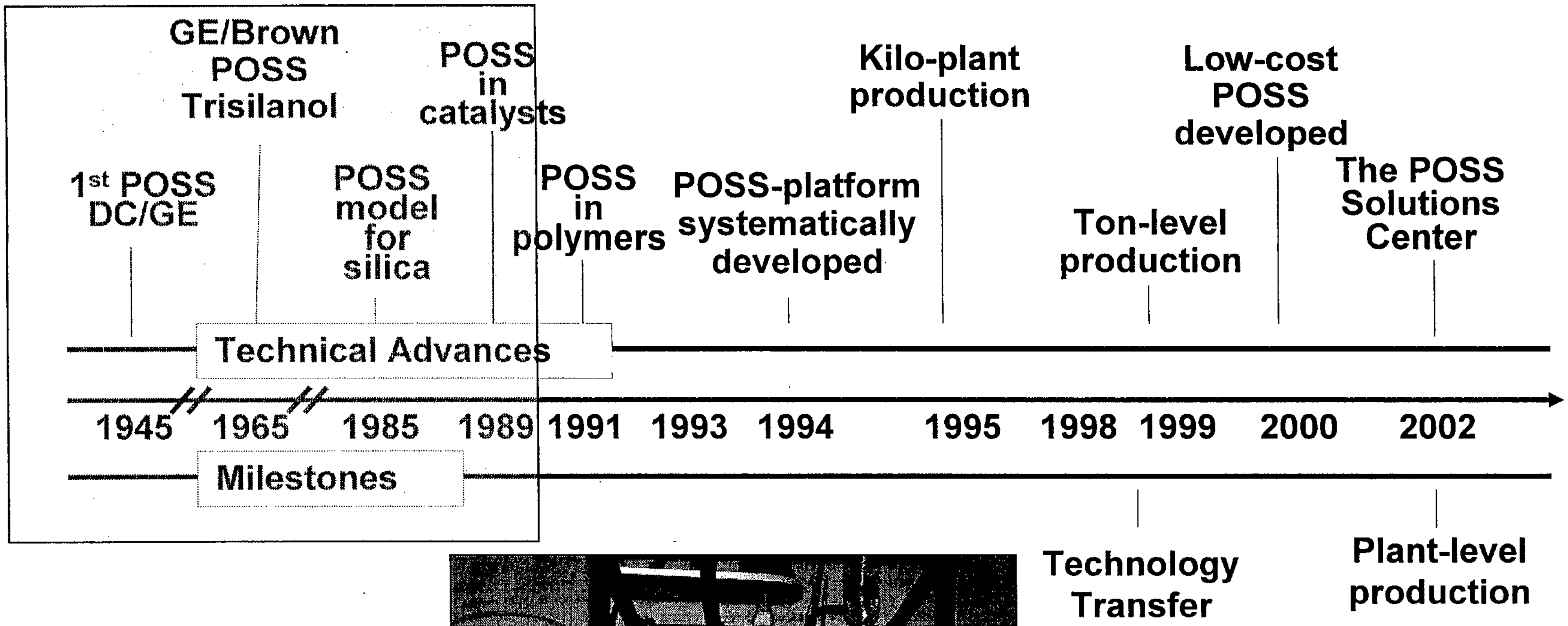
Commercialization: POSS®-Technology Timeline

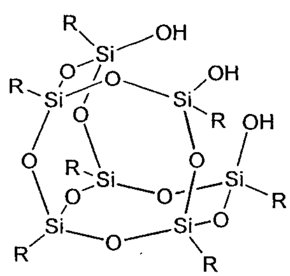


UCI — Chemistry

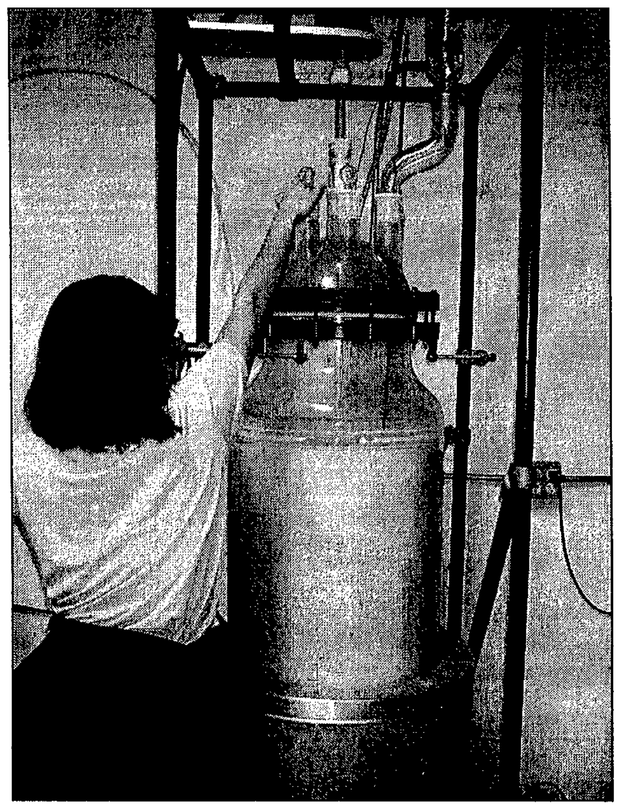
Air Force — Polymers

Hybrid Plastics — Commercial Solutions

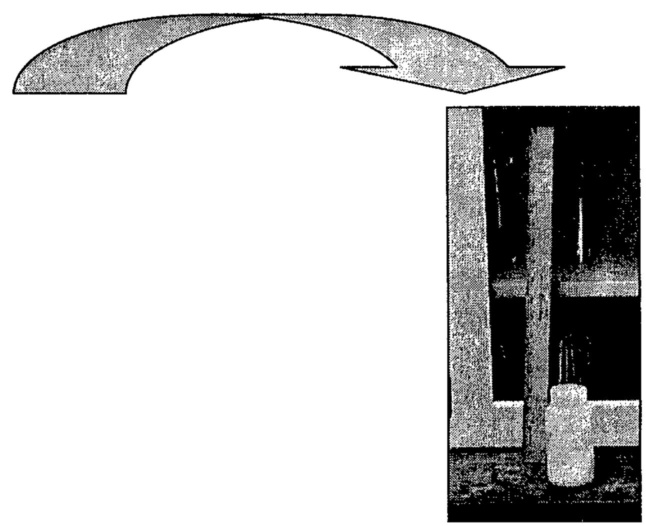


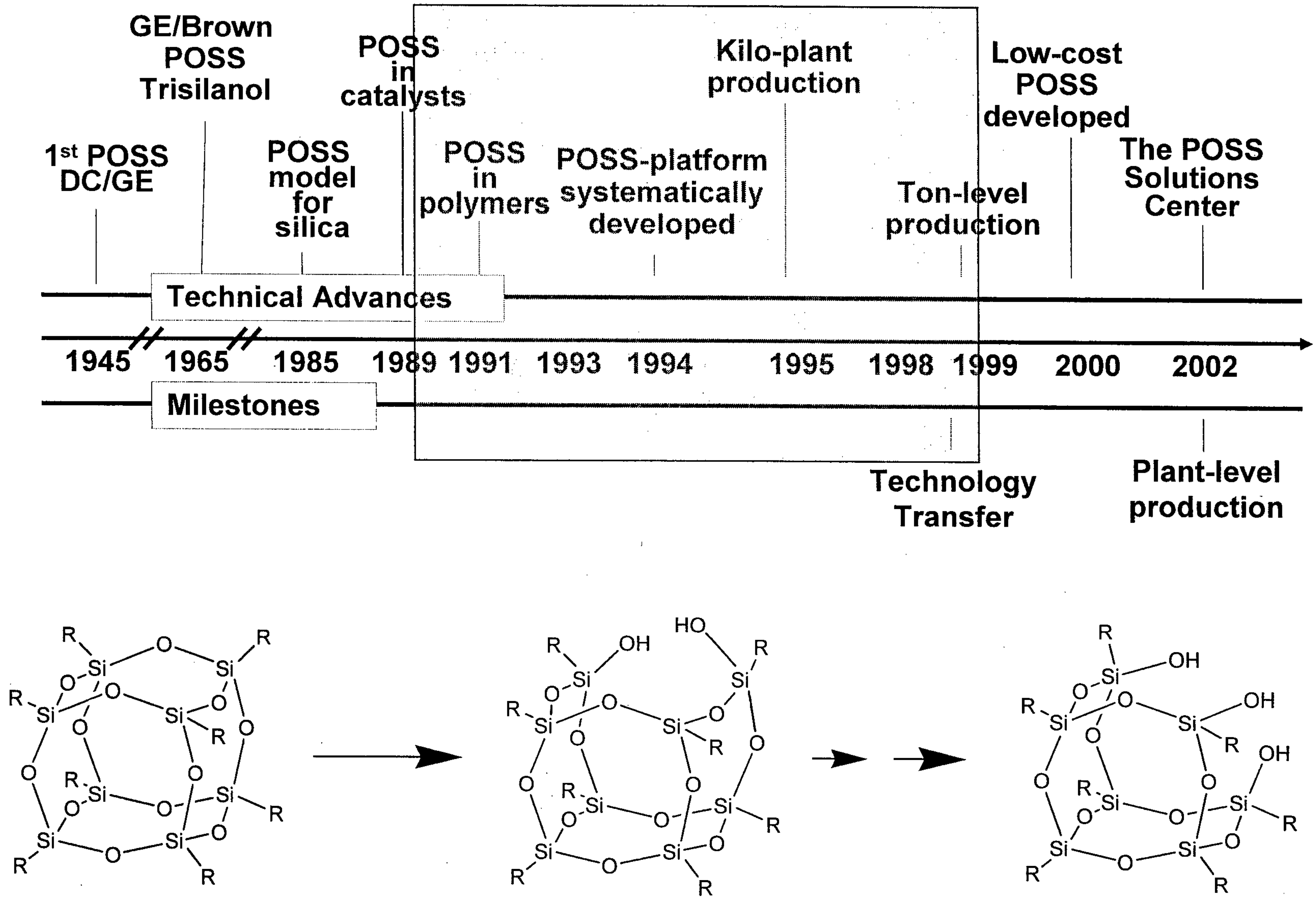


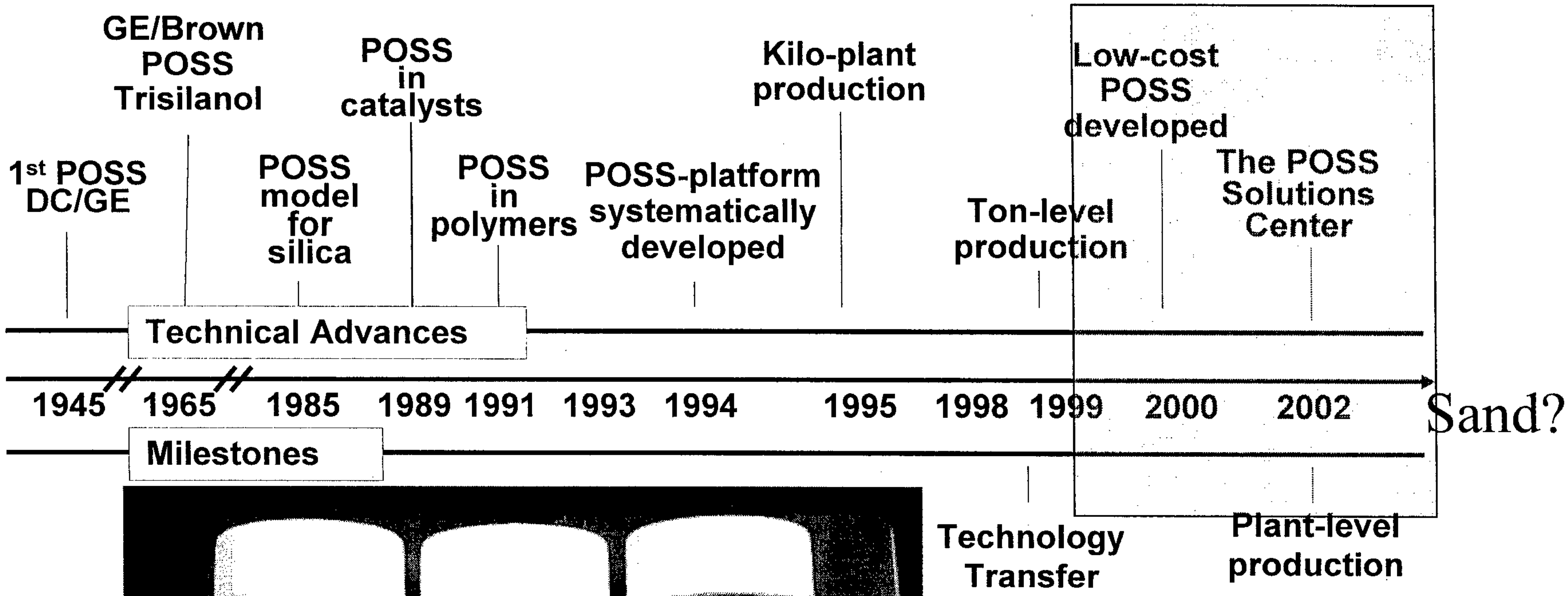
R = Cy

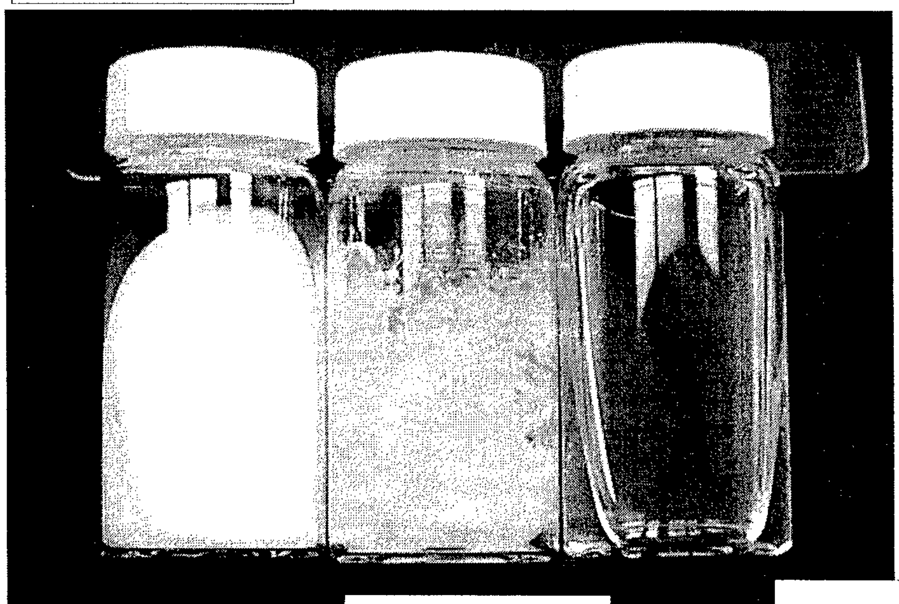


Reverse Scale-up?




1st POSS DC/GE
GE/Brown POSS Trisilanol
POSS model for silica
POSS in catalysts
POSS in polymers
POSS-platform systematically developed
Kilo-plant production
Ton-level production
Low-cost POSS developed
The POSS Solutions Center
Technical Advances
1945
1965
1985
1989
1991
1993
1994
1995
1998
1999
2000
2002
Milestones
Technology Transfer
Plant-level production
Si
O
R
OH
HO





Crystalline Solids

Wide melting range 24°C to 400°C+

Waxes

Liquids & Oils

Wide viscosity range 40cSt. to 400cSt

POSS® Applications

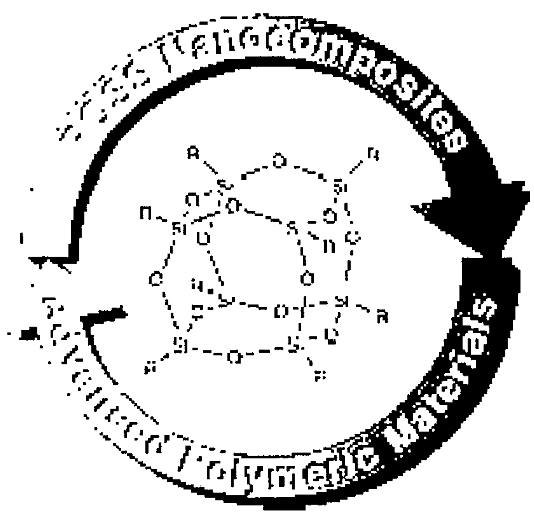
R&D Through Market Development

R&D Chemicals and Nanotechnology Markets

Aldrich Chemical Co.
Gelest Inc.
Hybrid Plastics

Monomers & Polymers

Aerospace
Electronics
Medical
Composites
Packaging



Blendable Agents

Viscosity Modifiers
Processing Aids
Fire Retardants
Performance Additives
Corrosion Resistance

Catalysis

Metathesis
Epoxidation
Ligands
Supports

Biology & Agriculture

Drug Delivery
Medical Prosthetics
Pharmaceuticals
Antifungal Agents